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Sampling for Water Quality

WATER QUALITY BRANCH, INLAND WATERS DIRECTORATE, OTTAWA, 1983

Canada

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Foreword

This first edition of "Sampling for Water Quality" describes field procedures for the sampling of surface water, precipitation, sediments and microbiological organisms, which are employed by the Water Quality Branch in its monitoring program to assess the quality of waters in Canada. It is intended as a reference manual for field staff and will also serve to document procedures currently in use by the Branch. The manual should also be of value to university, provincial, municipal and other investigators concerned with water quality.

As new procedures for fieldwork are developed and proved reliable through field testing, they will be published in subsequent editions of this manual.

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Comments on how to improve the contents and usefulness of this manual are most welcome. They should be sent to the Water Quality Branch at the same address as above.

N.H. James
Director General
Inland Waters Directorate

Contents

	Page
FOREWORD	iii
ABSTRACT	ix
RESUME	ix
ACKNOWLEDGMENTS	xi
DISCLAIMER	xi
1. INTRODUCTION	1
2. GENERAL WATER SAMPLING CONSIDERATIONS	2
2.1 Station Location	2
2.2 Observation-Based Samples	2
2.3 Field Quality Assurance	2
2.3.1 General Measures	2
2.3.2 Prevention of Sample Contamination	2
2.3.3 Field Quality Control	3
2.3.3.1 Bottle Blanks	3
2.3.3.2 Sampler Blanks	3
2.3.3.3 Filter Blanks	3
2.3.3.4 Field Blanks	4
2.3.3.5 Duplicate Samples (Splits)	4
2.3.3.6 Replicate Samples (Temporal)	4
2.3.3.7 Replicate Samples (Spatial)	4
2.3.3.8 Spiked Samples	4
3. COLLECTING SURFACE WATER SAMPLES	5
3.1 Types of Surface Water Samples	5
3.1.1 Grab Samples	5
3.1.2 Composite Samples	5
3.1.2.1 Sequential, or Time, Composite	5
3.1.2.2 Flow Proportional Composite	5
3.2 Collecting a Representative Water Sample	5
3.3 Field Equipment and Techniques	6
3.3.1 Grab Water Samplers	6
3.3.1.1 Depth Integrating Samplers	6

Contents (Cont.)

	Page
3.3.1.1.a Sampling Iron	6
3.3.1.2 Discrete Samplers	6
3.3.1.2.a Van Dorn Bottle	6
3.3.1.2.b Kemmerer Sampler	6
3.3.1.2.c Pumps	7
3.3.1.2.d Multiple Sampler	8
3.3.2 Dissolved Oxygen Sampler	8
3.4 Sampling Procedures as Influenced by Station Location and Season	8
3.4.1 Sampling Procedures from Bridges, Abutments, Boats and Aircraft	8
3.4.2 Sampling Procedures from Shores, Stream Banks and Wharves	9
3.4.3 Winter Sampling Procedures	9
3.5 Preparation for Field Trips	9
3.5.1 General Preparation	9
3.5.2 Bottle Washing and Preparation	10
3.5.3 Selection of Sample Volume	10
3.5.4 Checklist Prior to Field Trip	13
4. FIELD MEASURED PARAMETERS	14
4.1 pH Measurement	14
4.1.1 Procedure for Taking pH Measurements	14
4.1.2 pH Probe Maintenance	14
4.2 Conductivity Measurement	15
4.2.1 Procedure for Taking Conductivity Measurements	15
4.2.2 Conductivity Meter and Probe Maintenance	15
4.3 Dissolved Oxygen Measurement	15
4.3.1 Sampling	15
4.3.2 Dissolved Oxygen Meters	15
4.3.3 Winkler Analysis	16
4.3.3.1 Winkler Reagents	16
4.3.3.2 Winkler Procedure	16

Contents (Cont.)

	Page
4.3.4 Hach Method	17
4.3.4.1 Sample Collection	17
4.4 Temperature Measurement	17
4.4.1 In-field Procedure	17
4.5 Turbidity Measurement	17
4.5.1 In-field Procedure	18
4.6 General Summary of Field Procedures	18
5. RECORDING OF FIELD DATA	19
5.1 Station Location Description	19
5.1.1 Completion of Station Data Forms	19
5.2 Field Sheets	27
5.2.1 Completion of Field Sheets	27
5.2.1.1 Field Measurement Parameters	27
5.2.1.2 Instrument Calibration	30
6. FIELD FILTRATION AND PRESERVATION PROCEDURES	31
6.1 Filtration	31
6.1.1 General Apparatus Washing Procedures	31
6.1.2 Filtration Procedures	31
6.1.2.1 Particulate Carbon and Nitrogen	31
6.1.2.2 Chlorophyll a	32
6.2 Preservation Techniques	32
6.2.1 Chemical Addition	32
6.2.2 Freezing	32
6.2.3 Refrigeration	32
6.2.4 Practical Aspects of Preservation	32
7. SAMPLING FOR RADIOACTIVITY MEASUREMENTS	36
7.1 Sources of Radioactivity in Water	36
7.2 Collection and Preservation of Samples	36
8. SAMPLING FOR MICROBIOLOGICAL ANALYSIS	37
8.1 Sample Containers	37

Contents (Cont.)

	Page
8.2 Sample Preservation	37
8.3 Sampling Procedure	37
9. PRECIPITATION SAMPLING	38
9.1 Site Selection	38
9.2 Sample Collectors	38
9.3 Sample Collection Procedures	38
9.3.1 Definition of End of the Month	38
9.3.2 Daily Sample Handling Procedure	38
9.3.3 Sample Handling Procedure at the End of the Month	40
9.3.4 Prevention of Sample Contamination	40
9.4 Precipitation Sampling for Organic Analysis	40
9.4.1 Sample Handling Procedures	40
9.4.2 Precautions	40
9.5 Records of Precipitation Samples	40
9.5.1 Completing the Precipitation Sample History Form	40
10. SEDIMENT SAMPLING PROCEDURES	43
10.1 Sample Collection	43
10.1.1 Sample Integrity	43
10.1.2 Samplers	43
10.1.2.1 Suspended Sediment Samplers	43
10.1.2.2 Bottom Sediment Samplers	43
10.1.2.2.a Core Samplers	43
10.1.2.2.b Grab Samplers	43
10.1.3 Site Selection	45
10.1.3.1 Rivers	45
10.1.4 Sample Handling and Preservation	45
10.1.4.1 Suspended Sediment	45
10.1.4.2 Bottom Sediment	45
11. SHIPPING OF SAMPLES	46
11.1 Preparation for Shipping	46

Contents (Cont.)

Page

11.2 Shipping Labels..... 46

11.3 Labels on Individual Bottles..... 46

12. FIELD SAFETY 47

12.1 Safety Precautions when Sampling from Bridges..... 47

12.2 Safety Precautions when Sampling from Boats..... 47

12.3 Safety Precautions while Wading..... 47

12.4 Safety Precautions when Sampling through Ice..... 47

12.5 Safety Precautions when Handling Chemicals..... 48

12.6 Survival Kit and Rations..... 48

12.7 Proposed Water Quality Branch Field Safety Procedures..... 48

12.7.1 Proposed WQB Field Safety Procedures..... 48

GLOSSARY..... 50

REFERENCES..... 55

Tables

1. Washing Procedures and Containers for Water Samples..... 10

2. Washing Procedures and Containers for Bottom Sediments and Fish Tissue Samples..... 13

3. Specific Conductance of KCl Solutions at 25°C..... 15

4. NAQUADAT Codes for Types of Aquatic Media..... 20

5. Formats and Precision Codes for Sampling Locations..... 25

6. Sample Containers and Preservation for Constituents in Water..... 33

7. Sample Containers and Preservation for Constituents in Sediment..... 35

Illustrations

Page

Figure 1. Sampling Iron..... 6

Figure 2. Van Dorn Bottle..... 7

Figure 3. Kemmerer Sampler..... 7

Figure 4. Multiple Sampler..... 8

Figure 5. Dissolved Oxygen Sampler..... 8

Figure 6. Station Location Description Forms and Maps..... 21

Figure 7. Station Data Form..... 24

Figure 8. Field Sheet for Use with NAQUADAT or Similar Computer Systems..... 26

Figure 9. General Format for a Field Sampling Sheet..... 28

Figure 10. Sangamo Type A Precipitation Collector..... 39

Figure 11. Example of a Label for a Precipitation Sample..... 40

Figure 12. Example of a Completed Precipitation Sample History Form..... 41

Figure 13. Examples of Sediment Corers.... 44

Figure 14. Skipek Grab Sampler..... 45

Figure 15. Birge-Ekman Dredge..... 45

Abstract

This manual was designed to help the water quality investigator in the field. It outlines procedures which are currently practised and recommended by staff of the Water Quality Branch. The sample containers, preservatives and sampling procedures described are those commonly used for physical, chemical and bacteriological analyses.

Résumé

Le présent ouvrage a été conçu dans le but d'aider le chercheur qui prélève des échantillons d'eau aux fins d'analyses. On y trouve un résumé des méthodes utilisées et recommandées par la Direction de la qualité des eaux. Les contenants à échantillons, agents de conservation et méthodes d'échantillonnage qui y sont décrits, sont utilisés le plus communément pour effectuer les analyses physiques, chimiques et bactériologiques.

Acknowledgments

This field manual has drawn heavily on the sampling practices of all regional offices of the Water Quality Branch as well as procedures recommended by the U.S. Environmental Protection Agency (EPA) and the U.S. Geological Survey (USGS).

DISCLAIMER

The inclusion of trade names, commercial products or manufacturers in this manual is for illustration purposes only and does not constitute endorsement.

Introduction

The Water Quality Branch (WQB) of Environment Canada has the responsibility of assessing and providing information on the quality of Canada's inland waters. In 1972 the Branch published a small booklet outlining procedures for water quality sampling, "Instructions for Taking and Shipping Water Samples for Physical and Chemical Analyses." A second edition followed in 1973. The present publication is an updated and much expanded version of these instruction booklets.

Although the procedures recommended in this manual represent the best available, they may require adaptation under certain geological, hydrological or other environmental conditions. Water quality assessment on the whole will improve if the most reliable and accurate field techniques are employed. It is in this spirit that this field manual was prepared.

The manual will be updated to provide WQB staff with the latest procedures for the collection of water, precipitation, sediment and microbiological samples and field data.

General Water Sampling Considerations

The collection of water samples may seem a relatively simple task. However, more than the simple dipping of a container into water is required to obtain representative water samples and to preserve their integrity until they are analyzed in the laboratory. A representative sample can be easily obtained from rivers and lakes which are relatively homogeneous, whereas many bodies of water have significant spatial and temporal variations and the collection of a representative sample becomes much more complex. The procedures in this manual will aid investigators in collecting reliable, representative water samples.

The sections on water sampling equipment are not intended to be all-inclusive but rather to introduce common sampling equipment. Furthermore, instructions for operating sampling and field measurement equipment are not intended to replace those of the manufacturer but are to be considered as supplementary information. The sample containers, preservatives and sampling procedures described in this manual reflect those most widely used for physical, chemical and microbiological analyses by the Water Quality Branch. Shipping procedures and safety measures are also outlined.

2.1 Station Location

The location of sampling stations and the frequency of sampling must be outlined in the project design. They are established from the project objectives, and the spatial and temporal variability of the system. It is the responsibility of the field investigator to locate all sampling stations accurately. It is important to take the sample at exactly the same location each time. Only if the same location is consistently sampled can temporal changes in the water quality parameter levels be interpreted with confidence. Therefore, accurate station location descriptions must be prepared on the first visit to every sampling site, and these must be carefully followed by investigators on subsequent visits. An example of a format for the station location description is provided in Section 5.1.

2.2 Observation-Based Samples

In addition to locating sampling stations, field investigators are responsible for observing any unusual conditions which may indicate a need for additional water samples. Upon observing an unusual condition, such as an unusual colour or odour of the water, excessive algal growth, indications that foreign substances have entered the system (oil slicks, surface films, etc.) or fish kills, the field investigator should take samples in addition to those required by the project design. The type of samples and their preservation should be consistent with the type of analysis that the investigator thinks is warranted by the prevailing conditions. If additional samples are collected but not exactly at an established station, a new station location description should be accurately recorded. This information and additional samples will prove useful during the interpretive aspects of the study.

2.3 Field Quality Assurance

The Field Quality Assurance program is a systematic process which, together with the laboratory and data storage quality assurance programs, ensures a specified degree of confidence in the data collected for an environmental survey. The Field Quality Assurance program involves a series of steps, procedures and practices which are described in the following sections.

2.3.1 *General Measures*

(a) All equipment, apparatus and instruments should be kept clean and in good working condition by means of the techniques and practices given elsewhere in this manual.

(b) Records should be kept of all repairs to the instruments and apparatus and of any irregular incidents or experiences which may affect the success of the study.

(c) Conditions in the working area should be such that they encourage and maintain a completely safe environment (Section 12).

(d) It is essential that standardized and approved methodologies, such as those recommended in this manual, be used by field personnel. If any changes to the approved methods are made, they should be documented and experimental data obtained to ensure that the results which have been obtained are at least as good as before.

2.3.2 *Prevention of Sample Contamination*

The quality of data generated in a laboratory depends primarily on the integrity of the samples that arrive at the laboratory. Consequently, the field investigator must take the necessary precautions to protect samples from contamination and deterioration.

There are many sources of contamination; the following are some basic precautions to heed:

(a) Field measurements should always be made on a separate sub-sample, which is then discarded once the measurements have been made. They should never be made on the same water sample which is returned to the analytical laboratory for chemical analysis.

(b) Sample bottles, new or used, must be cleaned according to the recommended methods (see Tables 1 and 2 in Chapter 3).

(c) Only the recommended type of sample bottle for each parameter should be used (see Tables 1 and 2 in Chapter 3 and Tables 6 and 7 in Chapter 6).

(d) Water sample bottles should be employed for water samples only. Bottles that have been used in the laboratory to store concentrated reagents should never be used as sample containers.

(e) Before being used in the field, all preservatives must have been tested and the glassware spot-tested for cleanliness.

(f) Recommended preservation methods must be used (Section 6.2; Tables 6 and 7). All preservatives must be of analytical grade. They are usually provided and certified by the analytical laboratory.

(g) When preserving samples, the possibility of adding the wrong preservative to a sample or cross-contaminating the preservative stocks should be minimized by preserving all the samples for a particular group of parameters together.

(h) Solvent-rinsed Teflon or aluminum foil liners can be used to prevent contamination from the bottle caps of water samples which are to be analyzed for organic compounds.

(i) The inner portion of sample bottles and caps should not be touched with bare hands, gloves, mitts, etc.

(j) Sample bottles must be kept in a clean environment, away from dust, dirt, fumes and grime. Vehicle cleanliness is an important factor in eliminating contamination problems.

(k) Petroleum products (gasoline, oil, exhaust fumes) are prime sources of contamination. Spills or drippings (which are apt to occur in boats) must be removed immediately. Exhaust fumes and cigarette smoke can contaminate samples with lead and other heavy metals. Air conditioning units are also a source of trace metal contamination.

(l) Filter units and related apparatus must be kept clean, using procedures such as acid washes and soaking in special solutions, and should be wrapped in solvent-rinsed aluminum foil.

(m) Bottles which have been sterilized must remain sterile until the sample is collected. If the sterile heavy-duty paper or aluminum foil has been lost or if the top seal has been broken, discard the bottle.

(n) All foreign and especially metal objects must be kept out of contact with acids and water samples.

(o) Specific conductance should never be measured in sample water that was first used for pH measurements. Potassium chloride diffusing from the pH probe alters the conductivity of the sample.

(p) Samples must never be permitted to stand in the sun; they should be stored in a cool place; ice chests are recommended.

(q) Samples must be shipped to the laboratory without delay.

(r) The sample collector should keep his/her hands clean and refrain from smoking while working with water samples.

Additional sampling precautions related to water conditions at a particular location (shallow water) or season (winter) are discussed in Section 3.4.

2.3.3 *Field Quality Control*

Quality control is an essential element of a field quality assurance program. In addition to standardized field procedures, field quality control requires the submission of blank and duplicate samples to test the purity of chemical preservatives; to check for contamination, sample containers, filter papers, filtering equipment or any other equipment that is used in sample collection or handling; and to detect other systematic and random errors occurring from the time of the sampling to the time of analysis. Replicate samples must also be collected to check the reproducibility of the sampling. The timing and the frequency of blank, duplicate and replicate samples are established in the project design.

2.3.3.1 Bottle Blanks

Prior to a field sampling trip, one sample bottle for every ten of each type being used during the sampling trip should be selected at random, filled with ultrapure distilled water,* preserved in the same manner as field samples, and set aside for submission with the field samples for chemical analysis for the parameters of interest as "bottle blanks." This should detect any widespread contamination caused by the bottle washing process.

2.3.3.2 Sampler Blanks

Periodic "sampler blanks" consisting of ultrapure distilled water poured into, or permitted to pass through the sampler should be prepared and analyzed in the laboratory for the parameter(s) of interest.

2.3.3.3 Filter Blanks

If water samples are "field filtered" to determine the dissolved component of certain water quality constituents, the field filters should be pre-washed in the laboratory with a solution that can remove any contaminants which might affect the accuracy of measurement of the parameter of interest. Immediately

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* Ultrapure distilled water is obtained by passing distilled water through a Corning model AG-11 all-glass distillation unit and then through a Millipore Super Q Ultrapure Water System containing a prefilter cartridge, an activated carbon cartridge and a mixed bed deionization cartridge.

after washing, the filters should be sealed in plastic petri dishes for transport in the field. Filtering apparatus, such as funnels, should be pre-washed in the laboratory using the same procedure, and then sealed in Zip-Lock or Whirl-Pak polyethylene bags for transport in the field.

A daily "filter blank" should be prepared by passing a sample of ultrapure distilled water through one of the pre-washed filters in the filtration apparatus, preserving it in the same manner as the water samples, and then returning it to the laboratory for analysis for the parameter(s) of interest.

2.3.3.4 Field Blanks

Daily "field blanks" (one blank is suggested for every five water samples) should be prepared in the field at the end of each day's sampling by filling appropriate sample bottles with ultrapure distilled water, adding preservative in the same manner as it was added to the water samples, capping the bottles tightly, and transporting them to the laboratory in the same manner as the water samples. Field blanks need only be prepared for samples to be analyzed for parameters that require chemical preservation.

2.3.3.5 Duplicate Samples (Splits)

Duplicate samples are obtained by dividing one sample into two or more identical sub-samples. This should be done periodically to obtain the magnitude of errors owing to contamination, random and systematic errors, and any other variabilities which are introduced from the time of sampling until the samples arrive at the laboratory.

2.3.3.6 Replicate Samples (Temporal)

These are two or more samples taken at the same location sequentially at specified intervals over a specific period of time. They are taken to measure the uncertainty due to temporal variations of various parameters in the water body. The number and frequency of these samples are usually determined by a pilot study.

2.3.3.7 Replicate Samples (Spatial)

These are two or more samples taken simultaneously in a given predetermined cross section of the water body under study. They should be taken to measure the cross-sectional variations in the concentration of the parameters of interest in the system. The number and the exact location of these samples are usually determined by a pilot study.

2.3.3.8 Spiked Samples

At least once at each sampling point, control samples for each parameter being measured should be prepared by spiking a four-way split of a single water sample with three different levels of the parameter of interest, within the concentration range capability of the analytical method employed. The information gained from these control samples is used to reveal any systematic errors or bias in the analytical methodology, which is important in interpreting the data.

Collecting Surface Water Samples

The type of surface water sample collected is determined by a number of factors, such as:

(1) The objectives of the study, including the parameters of interest and the accuracy and precision needed.

(2) The characteristics of the system being studied, including the flow regime, climatic conditions, industrial inputs, groundwater infusions, tributaries, the homogeneity of the body of water, and the aquatic life present.

(3) The resources available, i.e., manpower, equipment and materials.

It is recommended that the design of the field sampling program be tested and assessed by a pilot project or in the initial rounds of sampling to ensure both its efficiency and effectiveness with respect to the objectives of the study. For example, assumptions about the temporal and spatial homogeneity of a river or lake can be tested by cross-sectional and vertical sampling, or the frequency of analysis of water quality parameters repeatedly below the detection limit can be reexamined. Other elements of the sampling program, such as ensuring that an adequate volume of water is collected or that the shipping of samples is adequate, can also be checked during the pilot project.

3.1 Types of Surface Water Samples

3.1.1 *Grab Samples*

A "discrete" grab sample is one that is taken at a selected location, depth and time, and then analyzed for the constituents of interest.

A "depth-integrated" grab sample is collected over a predetermined part or the entire depth of the water column, at a selected location and time in a given body of water, and then analyzed individually for the constituents of interest.

3.1.2 *Composite Samples*

A composite sample is obtained by mixing several discrete samples of equal or weighted volumes in one bottle, an aliquot of which is then analyzed for the constituents of interest.

The two main types of composite samples are:

3.1.2.1 Sequential, or Time, Composite

This composite is made up by:

(a) continuous, constant sample pumping or

(b) mixing equal water volumes collected at regular time intervals.

3.1.2.2 Flow Proportional Composite

This composite is obtained by:

(a) continuous pumping at a rate proportional to the flow,

(b) mixing equal volumes of water collected at time intervals which are inversely proportional to the volume of flow, or

(c) mixing volumes of water proportional to the flow collected during or at regular time intervals.

A composite sample provides an estimate of average water quality condition over the period of sampling.

3.2 Collecting a Representative Water Sample

For water quality sampling sites located on a homogeneous reach of a river or stream, the collection of depth-integrated samples in a single vertical may be adequate. For small streams a grab sample taken at the centroid of flow is usually adequate.

For sampling sites located on a nonhomogeneous reach of a river or stream, it is necessary to sample the channel cross section at the location at a specified number of points and depths. The number and type of samples taken will depend on the width, depth, discharge, the amount of suspended sediment being transported and aquatic life present. Generally, the more points that are sampled along the cross section, the more representative the composite sample will be. Three to five verticals are usually sufficient, and fewer are necessary for narrow and shallow streams.

The following general guidelines apply to the collection of a water sample:

(a) Do not include large nonhomogeneous particles, such as leaves and detritus, in the sample.

(b) Face the sampling apparatus upstream to avoid contamination. Sampling from the upstream side of a bridge enables the collector to see whether any floating material is coming downstream and aids in the prevention of contamination of the sample from paint chips or dirt from the road.

(c) Collect a sufficient volume to permit replicate analyses and quality control testing, if required. If not specified, the basic required volume is a summation of the volumes required for analysis of all the parameters of interest.

(d) Maintain accurate records on the field sampling sheets of possible sources of interference, environmental conditions and problem areas (Section 5.2.1).

3.3 Field Equipment and Techniques

3.3.1 *Grab Water Samplers*

Grab samplers may be divided into two broad categories: those appropriate for taking samples in which only non-volatile constituents are of concern and those for taking samples in which dissolved gases and other volatile constituents must be analyzed. Grab sampler types can also be divided into discrete (surface or specific depth) and depth integrating samplers. Both depth integrating samplers and discrete samplers may be used to collect water for the determination of non-volatile constituents. Also, a "multiple" sampler can be used for this purpose.

A grab sample may be taken using a "sampling iron," (Section 3.3.1.1.a) with an appropriate bottle; a Van Dorn bottle (Section 3.3.1.2.a); a Kemmerer style bottle (Section 3.3.1.2.b); or a pump type sampler (Section 3.3.1.2.c). Composite samples can be made from several grab samples or they can be obtained with special composite samplers (e.g. integrating samplers).

3.3.1.1 Depth Integrating Samplers

A depth-integrated sample may be taken by lowering an open sampling apparatus to the bottom of the water body and raising it to the surface at a constant rate so that the bottle is just filled on reaching the surface. This procedure will result in a sample which approximates a theoretical depth-integrated sample. The WQB sampling iron used for this purpose is briefly described here. Depth integration may not be possible in shallow streams where the depth is insufficient to permit integration.

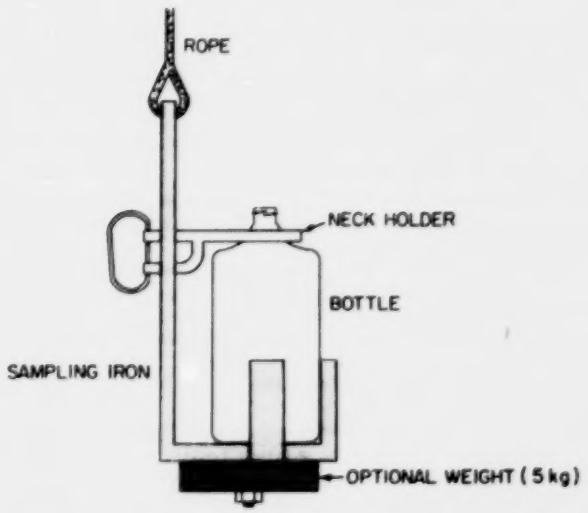

Figure 1. Sampling iron.

3.3.1.1.a Sampling Iron

This apparatus is a device which is made of iron and painted with a rust inhibitor. The weight of the sampler is approximately 2.7 kg (Fig. 1).

Typically, this design permits the use of a 2-L sample bottle when the bottle neck holder is in the uppermost position; smaller bottles may be used when the holder is located in lower positions.

The sample bottles are placed in the sampler and secured by the neck holder. In some cases, sampling irons may have provision for additional weights to ensure a vertical drop in strong currents. A depth-integrated sample is taken by permitting the sampler to sink to the desired depth at a constant rate and then retrieving it at approximately the same rate. The rate should be such that the bottle has just been filled when reaching the surface.

3.3.1.2 Discrete Samplers

Discrete samplers are used to collect water at a specific depth. An appropriate sampler is lowered to the desired depth, activated and then retrieved. Van Dorn, Kemmerer and pump type samplers are frequently used for this purpose.

3.3.1.2.a Van Dorn Bottle

The Van Dorn bottle is designed for sampling at a depth of 2 m or greater. The sampler, which is shown in its two configurations in Figure 2, is available in both polyvinyl chloride and acrylic plastic materials so that it may be used for general or trace metal sampling. Neoprene or silicone seals are available. The silicone seals are required for trace metal sampling. The end seals are made of semi-rigid moulded rubber or rigid machined plastic with gaskets. A drain valve is provided for sample removal. The horizontal configuration should be used when samples are taken at the bottom, at the sediment-water interface, or when samples are required from a narrow band of the depth profile (e.g. chemocline, thermocline). Sampler volumes from 2 to 16 L are available.

Although operation of the Van Dorn bottle varies slightly depending on its size and style, the basic procedure is the same:

(a) Open the sampler by raising the end seals.

(b) Set the trip mechanism.

(c) Lower the sampler to the desired depth.

(d) Activate a metal or rubber messenger to "trip" the mechanism that closes the end seals of the sampler.

(e) Transfer the water sample from the Van Dorn bottle to individual sample containers via the drain valve.

3.3.1.2.b Kemmerer Sampler

The Kemmerer style sampler is one of the oldest types of messenger-operated vertical samplers. It is commonly used in water bodies with a depth of 1 m or greater.

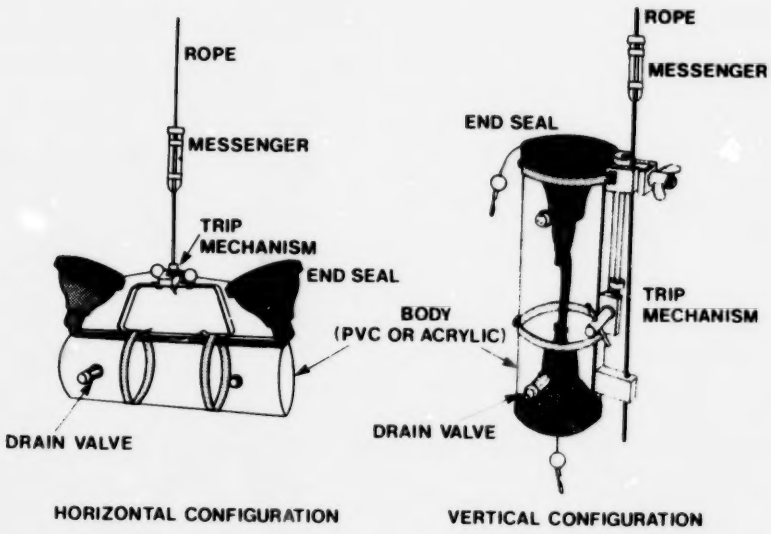

Figure 2. Van Dorn bottle.

The Kemmerer sampler, which is shown in Figure 3, is available in brass and nickel-plated brass for general water sampling. For trace metal sampling, Kemmerer samplers are made of polyvinyl chloride and acrylic plastic with silicone rubber seals. Both metal and plastic samplers are available in volumes ranging from 0.5 to 8 L.

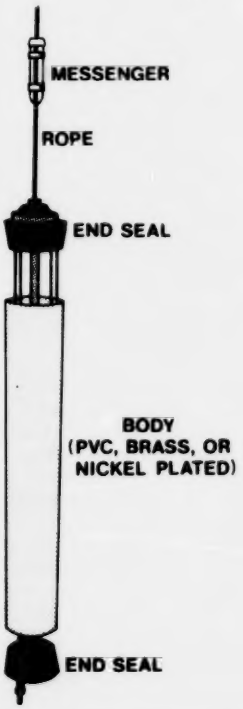

Figure 3. Kemmerer sampler.

The operation of the Kemmerer sampler is the same as that for the Van Dorn bottle.

3.3.1.2.c Pumps

Three types of pumps - diaphragm, peristaltic and rotary - are available to collect samples from specified depths. In general, diaphragm pumps are hand-operated; the peristaltic and rotary pumps require a power source and consequently they have limited field utility. Peristaltic pumps are not recommended for the collection of samples for chlorophyll analysis, as damage to the algal cells may occur. All pumps must have an internal construction that does not contaminate the water sample. Input and output hoses must also be free from contaminants.

The in-field procedure is:

(a) Place the input hose at the water depth specified by the sampling program. Take care not to pump up oil, algal mats, or other debris.

(b) Purge the pump and hoses with water from the station to be sampled before the actual water sampling begins.

(c) Each pump should be operated according to the instruction manual for that particular pump.

(d) Fill the type and number of sample bottles required at each station from the output hose.

Note: Take care not to contaminate the pump system. Do not permit the hoses to drag on the ground when the system is being transported.

3.3.1.2.d Multiple Sampler

A "multiple" sampler permits the simultaneous collection of several samples of equal or different volumes at a site. Each sample is collected in its own bottle. When the samples are of equal volume, information concerning the instantaneous variability between the replicate samples can be obtained. A multiple sampler is illustrated in Figure 4.

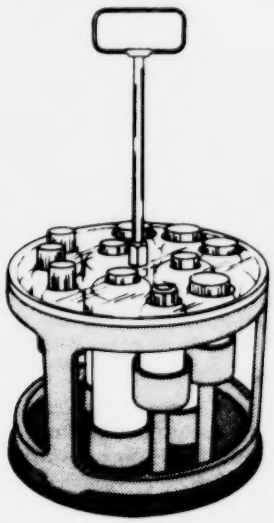

Figure 4. Multiple sampler.

The sampler may be altered to accommodate different sizes and numbers of bottles according to the requirements of specific programs. This may be done by changing cup sizes, length of cup sleeves and the configuration and size of openings in the clear acrylic top.

3.3.2 *Dissolved Oxygen Sampler*

A typical sampler for collecting samples for determining dissolved oxygen concentration and biochemical oxygen demand is illustrated in Figure 5.

The samples should be collected in narrow-mouthed biochemical oxygen demand (BOD) bottles that have bevelled glass stoppers to avoid entrapment of air in the samples. The procedure is outlined below:

(a) Place a 250- to 300-mL BOD bottle in the sampler and fasten the lid of the sampler in place, ensuring that the filling tube on the inside of the lid is positioned inside the BOD bottle.

(b) Lower the sampler into the water to the required depth and leave it there until air escaping from the sampler can no longer be seen.

(c) Retrieve the sampler and remove the lid. If bubbles are present in the bottle, tap the sides of the BOD bottle with the glass stopper. This procedure will release all trapped air bubbles. Place the special

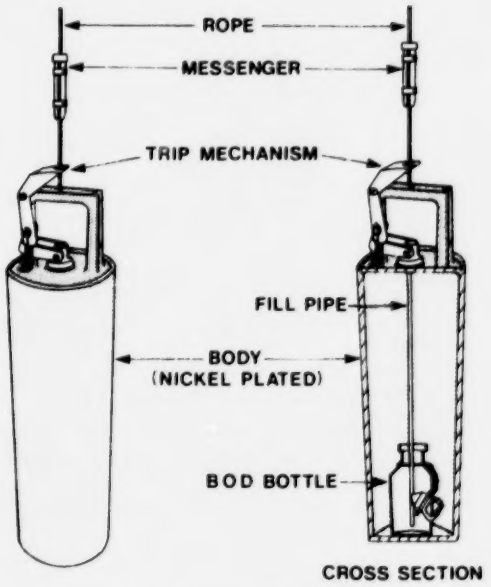

Figure 5. Dissolved oxygen sampler.

bevelled stopper in the BOD bottle and then remove the bottle from the chamber of the sampler.

Note: Sampling of shallow streams is not advisable with this sampler. If it is necessary to sample a shallow stream, gently tilt the bottle downstream, minimizing sample agitation (bubbling).

3.4 Sampling Procedures as Influenced by Station Location and Season

The use of proper sampling techniques and good judgment to obtain representative water samples is of utmost importance. In the field various sampling situations occur which require different sampling techniques. Situations in which water is shallow are handled in a manner and with apparatus different from that used at deep water sites. Field technicians must be equipped to handle these situations. In addition, special considerations and precautions must also be taken during periods of ice and snow (Section 3.4.3).

Since the fluvial characteristics of a sampling station can change with season, annual maximum and minimum flows and year-round accessibility should be considered when establishing a sampling station. When visiting an existing sampling station or when establishing a new site, the field investigator should take a variety of sampling equipment so that he or she is prepared for any situation. Some of the practical sampling considerations related to location and season of sampling are outlined in following sections.

3.4.1 *Sampling Procedures from Bridges, Abutments, Boats and Aircraft*

(a) Attach sufficient rope to permit the sampler to reach the required maximum depth. The other end

of the rope should be secured to a permanent fixture on the bridge, boat or aircraft.

(b) Ensure that all of the lines that are suspending the samplers (sampling irons, Van Dorn and Kemmerer types) remain in the vertical position to enable the accurate estimation of the depth of sample. Depending on the sampler used, weights may be added; the greater the stream velocity, the heavier the weight required. Every effort should be made to keep boats and float planes on location by anchoring or the use of the boat's motor.

(c) When sampling from a boat, sample from the upstream side; if sampling from a float aircraft, sample from the upstream and outer side of the pontoons to minimize the chance of contamination from engine oil leaks.

(d) When sampling, it is important that the sampling bottle not be permitted to touch the bottom of the river or lake to avoid contamination from stirred-up sediment. To prevent this it is useful to predetermine the water depth.

(e) Rinse the sampler three or four times with the water to be sampled unless the sampler contains a bottle which contains a preservative or is sterile.

(f) Obtain the required sample volume for field tests; then repeat the procedure to fill the remaining bottles.

(g) Carry out the required preservation procedures for each sample. See Section 6.2 on preservation techniques.

3.4.2 *Sampling Procedures from Shores, Stream Banks and Wharves*

A sampling iron is often used when water samples are collected from shores, stream banks and wharves.

(a) Insert an open clean sampling bottle into the metal holder provided, ensuring that the ring clamp is securely locked in the holder frame by a key ring or suitable pin. Attach sufficient rope to the holder to permit sampling at the desired depths. Secure the other end of the rope to a permanent fixture on the bank, wharf, etc. Sampling weights should be added as required, as dictated by stream velocity.

(b) Throw the bottle with holder well out into the stream. In the case of very shallow streams (approximately 0.5 m), the field investigator should collect the sample by hand, wading out if necessary, facing upstream and making sure not to contaminate the sample with sediment, debris and other floating materials.

(c) Pull the bottle and holder in quickly to prevent the bottle from touching or becoming snagged on the bottom of the stream.

(d) Rinse the sampling bottle three or four times with the water collected in step (c). It is important that the sample bottle be well rinsed with the water to be sampled before the sample is collected.

Note: If preservative has been added to the sample bottle prior to sampling or if the bottle is sterile, then do not rinse.

(e) Repeat steps (b) and (c).

(f) Rinse a beaker three or four times with the sampled water. Refill the beaker and carry out the field tests.

(g) After completion of field tests, repeat the sampling procedure to fill the remaining bottles required for laboratory tests.

(h) Carry out the required preservation procedures for each sample. See Section 6.2 on preservation techniques.

3.4.3 *Winter Sampling Procedures*

Sampling of ice and snow under winter conditions requires somewhat different techniques. *The safety precautions outlined in Sections 12.4 and 12.7.2.B must be followed.*

(a) Overlying snow should be removed from the ice surface to provide a suitable working area.

(b) Gas-powered augers are often used for drilling holes. Take extra care to avoid gas, oil and exhaust contamination of sampling equipment.

(c) Except in the case of shallow flowing streams, samples must not be taken from the hole in the ice but should be taken as a depth-integrated sample below the ice cover (Section 3.1.1).

(d) The hole in the ice must be cleaned of debris and ice chips; use a dip net or other "deslushing" device.

(e) Field measurements are not generally taken out on the ice but rather in the warmth of a vehicle, as meters tend to operate poorly in extremely cold conditions. An insulated box should be used and care taken to prevent samples from freezing in sub-zero temperatures.

3.5 Preparation for Field Trips

3.5.1 *General Preparation*

(a) Obtain specific instructions on sampling procedures.

(b) Prepare an itinerary according to the sampling schedule (see also Chapter 12).

(c) Prepare lists of required equipment and materials.

(d) Ensure that all sample bottles have been cleaned in accordance with standard procedures.

(e) Ensure that the laboratory has prepared the chemical reagents and standards needed for the trip.

(f) Prepare checklist (Section 3.5.4).

3.5.2 *Bottle Washing and Preparation*

Sample bottles are usually provided by the analytical laboratory. The recommended cleaning procedures are shown in Tables 1 and 2.

3.5.3 *Selection of Sample Volume*

The volume of sample required depends on the type and number of parameters to be analyzed, the analytical method, and the expected concentrations of the parameters in the water. Laboratory personnel will specify the sample volume required. The required sample volume can be determined by listing all of the parameters that are preserved in the same way, totalling the minimum volume for analysis and then multiplying by 2 for duplicate and 3 for triplicate analyses.

Table 1. Washing Procedures and Containers for Water Samples

Parameter(s) to be analyzed	Recommended container*	Washing procedure
Acidity Alkalinity Calcium Chloride Fluoride Hardness Magnesium pH Sodium Sulphate Colour Specific conductance Turbidity Nonfilterable residue Potassium Arsenic	1000 mL Polyethylene	Rinse three times with tap water, once with chromic acid,† three times with tap water, once with 1:1 nitric acid and then three times with distilled water in that order
Nitrogen; ammonia Nitrogen, nitrate nitrite Carbon, total organic Nitrogen, total	250 mL Polyethylene	Rinse three times with tap water, once with chromic acid,† three times with tap water and three times with distilled water in that order

* Teflon containers can also be used to replace either the recommended polyethylene or glass containers.

† Chromic acid - 35 mL saturated $Na_2Cr_2O_7$/1 L reagent grade conc. H_2SO_4.

‡ Chromic acid should not be used when the sample will be analyzed for chromium.

§ Ultrapure distilled water is obtained by passing distilled water through a Corning model AG-11 all-glass distillation unit and then through a Millipore Super Q Ultrapure Water System containing a prefilter cartridge, an activated carbon cartridge and a mixed bed deionization cartridge.

** Special grade acetone - pesticide grade when GC analysis to be performed, UV grade for LC analysis.

Table 1. Continued

Parameter(s) to be analyzed	Recommended container	Washing procedure
Phosphorus, total	50 mL Glass (Sovirel)	Rinse three times with tap water, once with chromic acid,† three times with tap water and three times with distilled water in that order
Aluminum Antimony Barium Beryllium Cadmium Chromium‡ Cobalt Copper Iron Lead Lithium Manganese Molybdenum Nickel Selenium Strontium Vanadium Zinc	500-1000 mL Polyethylene (depending upon number of metals to be determined)	Rinse three times with tap water, once with chromic acid,† three times with tap water, once with 1:1 nitric acid and then three times with ultrapure distilled water,§ in that order
Silver	250 mL Polyethylene (amber)	Rinse three times with tap water, once with chromic acid,† three times with tap water, once with 1:1 nitric acid and then three times with ultrapure distilled water,§ in that order
Mercury	100 mL Glass (Sovirel)	Rinse three times with tap water, once with chromic acid,† three times with tap water, once with 1:1 nitric acid and then three times with ultrapure distilled water,§ in that order
Organochlorinated pesticides and PCBs	1000 mL Glass (amber) with Teflon-lined cap	Rinse three times with tap water, once with chromic acid,† three times with

Table 1. Continued

Parameter(s) to be analyzed	Recommended container	Washing procedure
Organophosphorus	1000 mL Glass (amber) with Teflon-lined cap	organic-free water, twice with washing acetone, once with special grade** acetone ,twice with pesticide grade hexane and dry (uncapped) in a hot air oven at 360°C for at least 1 h
Pentachlorophenol	1000 mL Glass (amber) with Teflon-lined cap	Rinse three times with tap water, once with chromic acid,† three times with organic-free water, twice with washing acetone, once with special grade** acetone, twice with pesticide grade hexane and dry (uncapped) in a hot air oven at 360°C for at least 1 h
Phenolics	1000 mL Glass (amber) with Teflon-lined cap	Rinse three times with tap water, once with chromic acid,† three times with organic-free water, twice with washing acetone, once with special grade** acetone, twice with pesticide grade hexane and dry (uncapped) in a hot air oven at 360°C for at least 1 h
Phenoxy acid herbicides	1000 mL Glass (amber) with Teflon-lined cap	Rinse three times with tap water, once with chromic acid,† three times with organic-free water, twice with washing acetone, once with special grade** acetone, twice with pesticide grade hexane and dry (uncapped) in a hot air oven at 360°C for at least 1 h

Table 2. Washing Procedures and Containers for Bottom Sediments and Fish Tissue Samples

Parameter(s) to be analyzed	Recommended container*	Washing procedure
Aluminum Barium Beryllium Cadmium Chromium Cobalt Copper Iron Lead Lithium Manganese Mercury Nickel Zinc	Polyethylene bags (Whirl-Pak or Zip-Lock)	Use as shipped from manufacturer - no washing required
Organochlorinated pesticides and PCBs	Reynolds Aluminum small freezer trays, 11.4 cm x 14 cm	Rinse three times with tap water, three times with organic-free water, twice with washing acetone, once with special grade† acetone, twice with pesticide grade hexane and dry in a hot oven at 360°C for at least 1 h

* Teflon containers can also be used to replace either the recommended polyethylene or glass containers.
† Special grade acetone - pesticide grade when GC analysis to be performed, UV grade for LC analysis.

3.5.4 *Checklist Prior to Field Trip*

(a) Check and calibrate meters (pH, specific conductance, dissolved oxygen, turbidity) and thermometers.

(b) Replenish supplies of reagents for dissolved oxygen determinations as well as reagents for chemical preservation.

(c) Obtain fresh buffer solutions; pH values for the buffers should be close to the values expected in the field.

(d) Obtain KCl solution for pH probes.

(e) Obtain road maps, station location descriptions, field sampling sheets, sampling bottles, labels, samplers, preservation reagents, pipettes, equipment manuals, etc.

(f) Obtain writing materials, extra rope and a comprehensive tool box.

(g) Obtain charging cords if the equipment has in-field charging capabilities.

(h) Obtain distilled water and clean beakers for pH, blanks and buffer measurements.

(i) If field filtering is required, obtain filtering apparatus.

(j) If microbiological sampling is to be done, obtain sterile bottles and ice chests. Ice chests are recommended for all sample storage.

Field Measured Parameters

A number of parameters including pH, conductivity, dissolved oxygen, temperature and turbidity should be measured at the sampling site. Where possible, these measurements are taken *in situ*, but in all cases their values should be determined in the field as soon as possible after sample collection.

4.1 pH Measurement

The pH is a measure of the acidity or alkalinity of a solution. Neutral solutions have a pH of 7, acid solutions, a pH of less than 7, and alkaline solutions, a pH greater than 7.

The pH should be determined in the field, immediately after sample collection. Since pH can change rapidly and significantly soon after sample collection, it is not recommended that samples for this test be shipped to the laboratory. Optimally, pH is determined *in situ*, but if this cannot be done it can be determined by taking a water sample and measuring the pH as soon as possible.

There are many portable pH meters on the market today; the investigator should select the one that suits his needs best. Digital meters are preferable, since analog meters are sometimes difficult to read while taking *in situ* measurements (e.g. in a boat on rough water).

4.1.1 *Procedure for Taking pH Measurements*

Use the manual supplied with the instrument to calibrate, operate and troubleshoot the pH meter. In addition, the following should be considered:

(i) The instrument, including the battery, should be thoroughly checked and then calibrated in the laboratory, before it is taken to the field. The buffer solutions should periodically be checked in the laboratory.

(ii) In the field, the instrument should be recalibrated before each reading with appropriate buffer solutions and according to the instructions in the operating manual.

(iii) Adjust the temperature of the buffer solutions and electrodes by submerging the bottles of buffer and electrodes in the water sample. Extreme care must be taken to prevent the water from entering the buffer bottles or the filling hole of the reference electrode. An equivalent procedure is to measure the temperature of the buffer, to calibrate the meter and then to readjust the temperature compensation to the temperature of the sample.

(iv) If the electrodes have not been used recently, or have been allowed to dry for several days, they may require 10 to 20 min to stabilize. If the electrodes will not stabilize, try either a new reference or pH electrode. To keep the electrodes from drying out and ready for use, immerse the electrode tip in a rubber or plastic sack (such as the finger cut from a plastic glove) containing a few millilitres of buffer solution.

(v) In dry windy climates, a static charge tends to build on the plastic face of a pH meter, causing erratic movement of the indicator needle. Antistatic spray, such as that for phonograph records, sprayed on the meter face minimizes this problem.

(vi) Protect the meter from extreme temperature changes during measurement, as these affect the stability of the electronic system and, consequently, the precision of the measurement.

(vii) In cold waters, slow electrode response may result from the precipitation of the saturated filling solution in the reference electrode, thereby reducing the fluid contact. This can be remedied by permitting the filling solution to reach ambient temperature before filling the reference electrode. However, this will only remedy the situation for a few samples.

(viii) Before taking a measurement, thoroughly rinse electrodes and a glass or plastic beaker with sample water. Let the electrodes stand in the sample water for at least 2 min to stabilize before taking a reading. A reading, using a fresh sample, should be repeated until reproducible results ($\pm$0.1 pH units) are obtained.

4.1.2 *pH Probe Maintenance*

When combined electrode assemblies have been stored dry for a long period, the glass membrane should be soaked in a 3 mol/L KCl solution for 12 to 24 h before use. Meters may have a probe storage reservoir which should be filled with electrolyte. Glass electrodes that have not been conditioned before use may not stabilize properly and may require frequent recalibration.

If the pH meter shows a drift and the probe has been stored and correctly conditioned, the probe itself may require topping up with additional 3 mol/L KCl solution. Fill the probe according to specific assembly instructions. Frequent recalibration caused by electrodes with persistent drift may also be minimized by soaking the electrode in 0.1 *N* HCl or strong alkali.

The most common cause of trouble with combined electrode assemblies is a blockage in the diaphragm. If this occurs, as indicated by persistent drifting, soak the electrode in ammonium hydroxide. For optimum performance, electrodes should be soaked for 1 to 2 h in buffer prior to use, with buffers and samples equilibrated at the same temperature (use a constant temperature bath).

As with any piece of equipment, the meter and probe should be protected from dirt, freezing temperatures, and rough handling at all times.

4.2 Conductivity Measurement

Conductivity (specific conductance) is a numerical expression of a water's ability to conduct an electric current. Measured in microsiemens per centimetre (μS/cm), the conductivity depends on the concentration of ions in solution.

In situ measurements are preferable; if this is not possible, a sample is collected and measurement should be made as soon as possible, since the conductivity of a water sample may change with time. In most cases conductance readings are stable for months.

Conductivity is temperature dependent. If the conductivity measurement is not automatically temperature corrected, then the temperature at the time of measurement should also be recorded.

There are various conductivity meters available which may also have temperature and salinity determining capabilities. Since probes vary and cable lengths are optional, the investigator must select the equipment to meet the requirements of the sampling program.

4.2.1 *Procedure for Taking Conductivity Measurements*

The manual supplied with the conductivity meter should be followed for calibration, operation and trouble-shooting. Some general recommendations follow:

(i) The meter, including the battery, should be checked and calibrated before it is taken to the field.

(ii) The instrument should be recalibrated in the field before each reading.

(iii) To calibrate the instrument, KCl standard solutions with the specific conductance closest to the values expected in the field should be used (Table 3).

(iv) Do not use the same water sample in which the pH was measured to measure the specific conductance, as KCl diffuses from the pH electrode.

(v) Ensure that the probe does not touch the sides or bottom of the sample container.

(vi) Rinse sample containers and probe several times with the water sample.

(vii) Repeat the procedure with a fresh sample until reproducible conductivity readings are obtained, i.e. within ±5 %.

Table 3. Specific Conductance of KCl Solutions at 25°C

Concentration mol/L	Concentration g/L	Specific conductance μS/cm
0.0001	0.007456	14.94
0.0005	0.03728	73.90
0.001	0.07456	147.0
0.005	0.3728	717.8
0.00702	0.5234	1000
0.01	0.7456	1413
0.02	1.5912	2767

Note: This table has been modified from *Standard Methods for the Examination of Water and Wastewater* (1980).

4.2.2 *Conductivity Meter and Probe Maintenance*

Equipment for measuring conductivity must receive the same care and maintenance required by all sensitive instruments. Accurate readings require that the meter be protected from dirt, shocks and freezing temperatures.

4.3 Dissolved Oxygen Measurement

Dissolved oxygen (DO) should be measured *in situ* or in the field, as concentrations may show a large change in a short time if the sample is not adequately preserved. Even when the sample is preserved, as in a Winkler analysis, it is advisable to run the titrations within 3 to 6 h from the time the sample was taken. Dissolved oxygen concentrations may be determined directly with a DO meter or by a chemical method such as Winkler analysis. The method chosen will depend on a number of factors including the accuracy and precision required, convenience, equipment and personnel available and expected interferences. The methods discussed in this section are for DO meters, Winkler analysis and the Hach method. For very precise measurements the potentiometric method should be considered.

4.3.1 *Sampling*

Water samples for dissolved oxygen analysis should be collected using a specialized DO sampler (details of sampler are given in Section 3.3.3).

(a) Collect water samples using the dissolved oxygen sampler. Three samples must be taken. The recorded DO value will be the average of at least two readings that are within 0.5 mg/L of each other.

(b) Measure the dissolved oxygen concentration of the samples using a dissolved oxygen meter or Winkler chemical analysis.

4.3.2 *Dissolved Oxygen Meters*

Dissolved oxygen meters can be used to measure dissolved oxygen concentrations *in situ*, in the field or in the laboratory. The procedure is as follows:

(i) calibrate the instrument by placing the probe in a water sample of known dissolved oxygen concentration, i.e., determined by the Winkler method, or in a freshly air-saturated water sample of known temperature. With the latter method calibration can be done by using a table listing the solubility of oxygen in water at different temperatures and pressures.

(ii) Place the probe in the solution to be measured and agitate the probe. Let the probe come to temperature.

4.3.3 *Winkler Analysis*

This method can be used to determine dissolved oxygen concentrations in the field or laboratory.

4.3.3.1 Winkler Reagents*

Manganous sulphate solution - Dissolve 365 g manganous sulphate monohydrate, $MnSO_4 \cdot H_2O$, or 400 g manganous sulphate dihydrate, $MnSO_4 \cdot 2H_2O$, or 480 g manganous sulphate tetrahydrate, $MnSO_4 \cdot 4H_2O$, in freshly boiled distilled water and dilute to 1 L. A magnetic stirrer will speed up dissolution. Store in a glass bottle.

Alkaline-iodide-azide solution - Dissolve 400 g sodium hydroxide pellets, NaOH, in 500 mL of freshly boiled distilled water. Add the sodium hydroxide in small increments and dissolve each addition before proceeding. Add 900 g sodium iodide, NaI, while the solution is still hot. Dissolve 10 g sodium azide, NaN_3, in 40 mL distilled water. Add the sodium azide solution to the first solution and dilute, if necessary, to 1 L.

Sulphuric acid, concentrated reagent grade.

Potassium bi-iodate, 0.01 *N* - Dry a suitable amount of primary standard grade bi-iodate, $KH(IO_3)_2$, at 105°C for 1 h. After cooling in desiccator, weigh out 0.3249 g and dissolve in distilled water. Dilute to 1 L and store this solution in a tightly stoppered dark glass bottle.

Starch indicator solution - Add 30 g of soluble starch to 1 L of glycerol. Heat the mixture to 180°C, maintaining the temperature until the solution is transparent.

Sodium thiosulphate solution, 0.018 *N*, 1 mL = 1.5 mg DO - Dissolve 4.8 g sodium thiosulphate pentahydrate, $Na_2S_2O_3 \cdot 5H_2O$, and 0.1 g sodium carbonate, Na_2CO_3, in 1 L of distilled water. Add a few drops of chloroform as a preservative.

Standardization procedure - Fill a 250-mL BOD bottle with water (not chlorinated tap water). Add 1.0 mL alkaline-iodide-azide reagent, replace the stopper, and mix. Add 1.0 mL concentrated sulphuric acid and mix. Add 1.0 mL manganous sulphate and mix again. Transfer 100 mL of solution from the BOD bottle to an Erlenmeyer flask and add 10.0 mL potassium bi-iodate solution. Let the mixture stand for at least 2 min in the dark, then titrate the liberated iodine with thiosulphate solution to a very pale straw colour. Add 4 drops of starch solution. Continue the titration until the blue colour just disappears.

Normality (N_1) of thiosulphate is obtained from the expression

$$N_1 = N_2 \times V_2/V_1$$

where N_1 = normality of sodium thiosulphate,
V_1 = volume of thiosulphate,
N_2 = normality of the standard bi-iodate solution,
V_2 = volume of potassium bi-iodate solution. (This is always 10.0 mL.)

4.3.3.2 Winkler Procedure

Remove stopper from the BOD bottle and add, using pipettes placed just below the water surface, 1.0 mL of manganous sulphate reagent followed immediately by 1.0 mL of alkaline-iodide-azide solution. Re-stopper the bottle at once and mix the contents by shaking vigorously for at least 20 s or until the precipitated manganous and manganic hydroxide is evenly dispersed. No air bubbles should be trapped in the bottle. Let it stand for about 2 to 3 min, then shake the bottle again. Allow the precipitate in the sample to settle at least one third of the way down the bottle (about 10 to 20 min required).

Add 1.0 mL concentrated, reagent grade H_2SO_4 by permitting the acid to run down the neck of the bottle. Re-stopper the bottle and shake gently until the dissolution of the precipitate.

By means of a pipette transfer 100 mL of solution from the BOD bottle to an Erlenmeyer flask.

Titrate at once with thiosulphate solution to a very pale straw colour. Add 4 drops of starch solution. Continue the titration until the blue colour just disappears.

Calculate the dissolved oxygen content of the sample as follows:

$$\text{mg/L DO} = \frac{N \times V \times 8 \times 100}{99.3}$$

where N = normality of sodium thiosulphate solution,
V = volume of sodium thiosulphate solution used in the titration.

This method is not applicable under the following conditions:

(i) Samples containing more than 1 mg/L of ferrous iron. However, if 1 mL fluoride solution is added before acidifying the sample and there is no delay in titration, the method can be used in the presence of up to 100-200 mg/L ferrous iron. The potassium fluoride solution is made by dissolving 40 g $KF \cdot 2H_2O$ in distilled water and diluting to 100 mL.

(ii) Samples containing sulphite, thiosulphite, polythionate, free chlorine or hypochlorite.

(iii) Samples with high concentrations of suspended solids.

(iv) Samples containing other oxidizing or reducing materials.

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* As described in the *Analytical Methods Manual* (Water Quality Branch, 1981).

In instances where this method is not applicable, a DO probe should be used.

4.3.4 *Hach Method*

The Hach* method is used for determining dissolved oxygen concentrations in the field. It involves the same chemical reactions as Winkler titration. The reagents, except the titrant, which is PAO (phenylarsine oxide), not thiosulphate solution, are contained in individual "powder pillows" premeasured for the required concentrations.

This method can be used when results within ±0.5 to 1.0 mg/L of the true value are sufficient for the purpose of the study.

The kit contains a Hach dissolved oxygen sampler, 300-mL BOD bottle, 60-mL BOD type bottle, a vial and a small dispensing bottle containing the PAO titrant, as well as pillows of reagents in the dry granular form. Starch, which is not supplied with the kit, must be obtained to assist in the detection of the end point.

4.3.4.1 Sample Collection

(i) After the removal of the glass stopper, place the 60-mL DO sample bottle in the sampler.

(ii) Replace the sampler lid, with the sample inlet tube extending into the bottle.

(iii) Lower the sampler by the chain; then pull out the stopper at the desired depth.

(iv) When air bubbles stop rising from the sampler, raise the sampler by the chain and carefully remove the sample bottle.

(v) Add the contents of one pillow of Dissolved Oxygen I Powder (manganous sulphate) and the contents of a Dissolved Oxygen II Powder (alkaline-iodide-azide) pillow. Stopper the bottle carefully so that air is not trapped in the bottle (see Note A below). Grip the bottle and shake vigorously (see Note B below). A flocculant precipitate will be formed. If oxygen is present, the precipitate will be brownish orange.

(vi) Let the sample stand until the floc has settled halfway and leaves the upper half of the bottle clear. Again shake the bottle and let it stand until the upper half of the bottle is clear.

(vii) Remove the stopper and add the contents of one Dissolved Oxygen III Powder pillow (dry acid). Carefully re-stopper (see Note A) and shake to mix. The floc will dissolve and a yellow colour will develop if oxygen is present. This is the prepared sample.

(viii) Pour off the contents of the DO bottle until the level just reaches the 30-mL mark on the bottle.

(ix) While swirling the sample to mix, quickly add PAO by drops, counting each drop until the samples change to a pale straw colour. Add a few drops of starch solution. Continue adding PAO until the colour changes from blue to colourless. Each drop is equal to 0.2 mg/L dissolved oxygen.

Notes:

A. It is difficult to stopper the DO bottle without getting air bubbles trapped in the bottle. To avoid air bubbles, tip the DO bottle slightly and insert the stopper with a quick thrust. This will force air bubbles out. If air bubbles are trapped in the DO bottle in steps (v) or (vii), the sample should be discarded and the test started over.

B. A small amount of powdered reagent may remain stuck to the bottom of the DO bottle at this point, but this will not affect the test.

C. Do not permit the PAO solution to stand in direct sunlight, as it is decomposed by ultraviolet radiation.

D. When the water is saturated with dissolved oxygen, small bubbles of oxygen coming out of solution in the 60-mL bottle will cause an air bubble to form at the stopper. This is unavoidable; proceed with instructions as usual.

4.4 Temperature Measurement

Temperature should optimally be measured *in situ*, as the temperature of any water sample will readily equilibrate with its surroundings.

Temperature measurements may be taken with alcohol-toluene, mercury-filled or dial type thermometers. In addition some meters, such as those used to measure dissolved oxygen and specific conductance, have temperature measuring capabilities. Temperature values at specified depths can be measured *in situ* if adequate cables and probes are available. Otherwise, temperatures must be measured immediately in the field after the water sample has been taken from the required location and depth.

4.4.1 *In-field Procedure*

If a thermometer is used:

(a) Rinse the thermometer by pouring a portion of the water sample over it.

(b) Immerse the thermometer in the sample for approximately 1 min or until the reading stabilizes. Do not place the thermometer in any of the sample bottles being shipped to the laboratory.

(c) Record the value in degrees Celsius on the field sheet.

4.5 Turbidity Measurement

Turbidity is a measure of the suspended sediment such as clay, silt, organic matter, plankton and microscopic organisms in a water sample. Whenever possible, turbidity should be measured in-field. Otherwise, the

* Hach Chemical Company, Ames, Iowa, U.S.A.

turbidity is measured at the laboratory. The field measurement is preferable, since some of the particulate matter will settle or adhere to the container wall during transportation. Furthermore, changes in the pH of the sample may cause the precipitation of carbonates and humic acids, affecting the turbidity of the sample. When the analysis cannot be done immediately, the sample should be stored in the dark and for not more than 24 h.

Turbidity can be measured by visual methods (in Jackson Turbidity Units or JTU) or nephelometric units (in Nephelometric Turbidity Units or NTU). Nephelometric methods are preferred on account of their greater precision, sensitivity and application over a wide turbidity range.

4.5.1 *In-field Procedure*

(a) To operate the turbidity meter, follow the manual for the instrument.

(b) Prepare calibration curves for each range of the instrument by using appropriate standards.

(c) Test at least one standard in each range to be used.

(d) Make certain that the turbidity meter gives stable readings in all sensitivity ranges used.

(e) Keep sample tubes clean both inside and out; replace them when they become scratched or etched. Do not handle the tubes in the region where the light beam enters them.

(f) Shake the sample vigorously before analysis. Readings should always be made after the same time period following the homogenizing of the sample (e.g. 10 s) to ensure uniform data.

(g) It is important to pour off the sample quickly and to measure the turbidity of the sample in triplicate.

4.6 General Summary of Field Procedures

Regardless of the specific parameters of interest, a routine should be followed at each sampling station. The following is a general summary of procedures to be followed at each station.

(a) Calibrate meters.

(b) Standardize sodium thiosulphate when using Winkler analysis for dissolved oxygen.

(c) Run field or *in situ* measurements for pH, conductivity, dissolved oxygen, temperature and turbidity.

(d) Rinse all bottles with sampled water except for those which contain preservatives or those used for dissolved oxygen and bacteria analyses.

(e) Collect and preserve samples according to this instruction manual.

(f) Complete field sheet accurately according to this instruction manual.

(g) Put bottles in appropriate shipping containers.

(h) Label boxes and complete field sheets with all required information.

Recording of Field Data

For water quality data to be retrievable from a data system, the sampling location, sampling date, parameter measured and value must be associated.

5.1 Station Location Description

The importance of an accurate written description of each station location and the conditions under which the samples are collected cannot be overemphasized. In fact, interpretation of water quality data may be limited by the accuracy of such descriptions. It is therefore recommended that a precise format, such as that shown in Figure 6, be used.

An accurate description of the sampling location includes distances to specific reference points. It is important that these reference points be long-standing and clearly identified. For example, "5 m NW of the willow sapling" is a poor location designation for a long-term sampling program. An example of a correct description would be (see also Fig. 6) "30 m downstream from Lady Aberdeen Bridge (Highway 148), between Hull and Pointe Gatineau, and 15 m off the pier on the left side looking downstream."

The date the station was first established (sampled) must also be entered.

Station Description - Enter descriptions of the water body above and below the sampling station, describe the banks on either side of the water body, and the bed material, if known. Description of the water body should include any irregularities in morphology affecting flow or water quality. These irregularities may include a bend in a river, widening or narrowing of the channel, presence of an island, rapids or falls, or the entry of a tributary near the sampling station. Description of the banks should mention slope, bank material and extent of vegetation. Bed or sediment material may be described as rocky, muddy, sandy, vegetation-covered, etc. Station location descriptions should mention seasonal changes that may interfere with year-round sampling.

Observations - Enter any additional information about conditions, either natural or man-made, which may have a bearing on water quality.

Detailed Sketch of Station Location - Sketch the location of the sampling station (including distances expressed in suitable units) with respect to local landmarks and permanent reference points. A sample sketch is provided in Figure 6.

Map - Include a map which locates the sampling station on a larger scale with respect to roads, highways and towns. The combination of the map and the sketch of the station location should provide complete location information. An investigator travelling to the site for the first time will therefore have enough information to locate the sampling station confidently and accurately.

5.1.1 *Completion of Station Data Forms*

To store data in NAQUADAT,* each new sampling location requires the completion of a "Station Data" form, as illustrated in Figure 7.

The information required to complete this form, as outlined in the *NAQUADAT Guide to Interactive Retrieval* (Whitlow and Lamb, 1982), is as follows:

(1) Station number.

(2) Location as latitude and longitude or UTM (Universal Transverse Mercator) coordinates.

(3) Narrative description.

(4) Reference station.

(5) Station parameters.

Although only the first and either the second or third items are mandatory, the practice in the Water Quality Branch is to require that at least items (1) to (3) be present before a new station is accepted in NAQUADAT.

The following is a more detailed description of the fields referred to above.

1. Station Number - a 12-digit alphanumeric data processing code, which is the key element in storing and retrieving data on the system. The station number is composed of several subfields:

   Type of Water - a two-digit numerical code indicating the type of water sampled at any given location, such as streams, rivers, lakes or precipitation. The meaning of this code has been extended to include other types of aquatic media, and a list of all currently assigned codes is given in Table 4, or can be found in the *NAQUADAT Guide to Interactive Retrieval* (Whitlow and Lamb, 1982).

* NAQUADAT - the National Water Quality Data Bank, which is operated by the Water Quality Branch, Department of the Environment.

Province - a two-digit alphabetical code. The following codes have been assigned to the provinces and territories (in alphabetical order).

Alberta	AL
British Columbia	BC
Manitoba	MA
New Brunswick	NB
Newfoundland	NF
Northwest Territories	NW
Nova Scotia	NS
Ontario	ON
Prince Edward Island	PE
Quebec	QU
Saskatchewan	SA
Yukon Territory	YT
Alaska	AK
Other U.S.	US

Table 4. NAQUADAT Codes for Types of Aquatic Media

Type	Code	Subtype	Code
Surface water	0	Stream - channel	0
		Lake	1
		Estuary	2
		Ocean - sea	3
		Pond	4
		Impounded reservoir	5
		Harbour	6
		Ditch	7
		Runoff	8
		Unknown	9
Groundwater	1	Well - sump	0
		Spring	1
		Piezometer well	2
		Tile drain	3
		Bog	4
		Household tap	8
		Unknown	9
Waste - treated and untreated	2	Industrial	0
		Municipal	1
		Mining	2
		Livestock waste	3
		Unknown	9
Precipitation	3	Rain	0
		Snow	1
		Ice (precipitated)	2
		Mixed precipitation	3
		Dry fallout	4
Treated supply	4	Municipal	0
		Industrial	1
		Mining	2
		Private (individual)	3
		Other communal works	4
		Municipal distribution	5
		Municipal treatment plant (intermediate)	6
		Treatment residue or sludge	7
		Other	9
Sediments, soils	5	Stream channel	0
		Lake bottom	1
		Stream bank	2
		Lake bank	3
		Contaminated by soil	4
		General soil	5
		Effluent irrigation soil	6
		Sludge conditioned soil	7
		Other	8
Industrial wastewater	6	Storm water	0
		Primary influent	1
		Primary effluent	2
		Final effluent	3
		Sludge	4
		Special problem	5
		Other	6
Municipal wastewater	7	Raw	0
		Primary lagoon effluent	1
		Secondary lagoon effluent	2
		Conventional primary effluent	3
		Conventional secondary effluent	4
		Advanced wastewater treatment effluent	5
		Disinfected effluent	6
		Raw sludge	7
		Digested sludge	8
		Other	9
Miscellaneous wastewater	8	Raw	0
		Primary lagoon effluent	1
		Secondary lagoon effluent	2
		Conventional primary effluent	3
		Conventional secondary effluent	4
		Advanced wastewater treatment effluent	5
		Disinfected effluent	6
		Raw sludge	7
		Digested sludge	8
		Other	9
Aquatic biota	9	To be categorized later (e.g., fish, phytoplankton, benthos, macrophytes, periphyton, zooplankton)	

Source: Whitlow and Lamb (1982).

DOE, INLAND WATERS DIRECTORATE, WATER QUALITY BRANCH

STATION LOCATION DESCRIPTION

REGION QUEBEC

PROVINCE QUEBEC BASIN OTTAWA RIVER

STATION DATA

Type	Prov.	Basin	Sub-Basin	Sequent		
00	04	02	L	11	003	600

Latitude (N+)				Longitude (W+)				Pr
S	Deg	Min	Sec	S	Deg	Min	Sec	
	45	27	25.00		075	42	02.00	5

UTM Zone	Easting	Northing (N+)	Pr
	S 0	S	

STATION LOCATION

ON GATINEAU (Reservoir / Stream / Lake / (River))

at LADY ABERDEEN BRIDGE near PTE. GATINEAU Prov. QUE.

Located in _____ Sec. _____ Tp _____ Rge _____

Established APRIL 1978

Distance from base to station 1.5 Km

Distance from station to site of analysis 17 Km

Location of station with respect to towns, bridges, highways, railroads, tributaries, islands, falls, dams, etc.:

30M DOWNSTREAM OF LADY ABERDEEN BRIDGE (HIGHWAY 148) BETWEEN HULL AND POINTE GATINEAU AND 15M OFF PIER ON LEFT SIDE (LOOKING DOWNSTREAM)

Description and location of nearby hydrometric installations

BASKATONG DAM ABOUT 190 KM UPSTREAM
FARMERS RAPIDS ABOUT 25 Km UPSTREAM

SPECIMEN

Figure 6. Station location description forms and maps.

STATION DESCRIPTION

DIRECTION OF FLOW:

SOUTH EAST

DESCRIPTION OF CHANNEL ABOVE STATION:

PERMANENT LOG BOOM ON RIGHT, GRADUAL CURVE TO LEFT.

DESCRIPTION OF CHANNEL BELOW STATION:

GRADUAL WIDENING BEFORE EMPTYING INTO OTTAWA R.; MAIN CURRENT ON LEFT, SLIGHT BACKWATER ON RIGHT.

DESCRIPTION OF LEFT BANK:

APPROX. 3M DROP TO RIVER, SLOPE ALLOWS ONLY SHRUBBY VEGETATION.

DESCRIPTION OF RIGHT BANK:

EDGE OF PARKLAND; GENTLE SLOPE

BED: rocky, gravel, sandy, clean, vegetated.

PROBABLY WOOD CHIPS, MUDDY

APPROXIMATE DIMENSIONS AND DESCRIPTIONS OF LAKES AND/OR RESERVOIRS

NONE

OBSERVATIONS

NATURAL CONDITIONS AND/OR CONTROL INSTALLATIONS WHICH MAY AFFECT FLOW REGIMES:

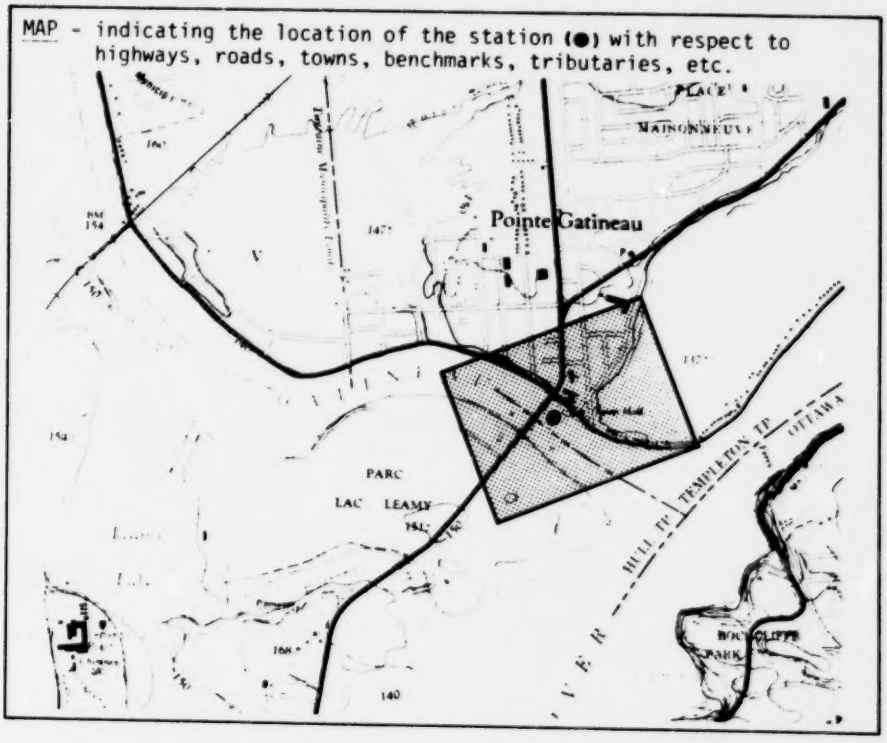
BASKATONG DAM

FARMERS RAPIDS

SOURCES OF CHEMICAL OR PHYSICAL INPUTS:

LOGS, LOCAL SEWAGE INPUT.

Figure 6. Station location description forms and maps (continued).



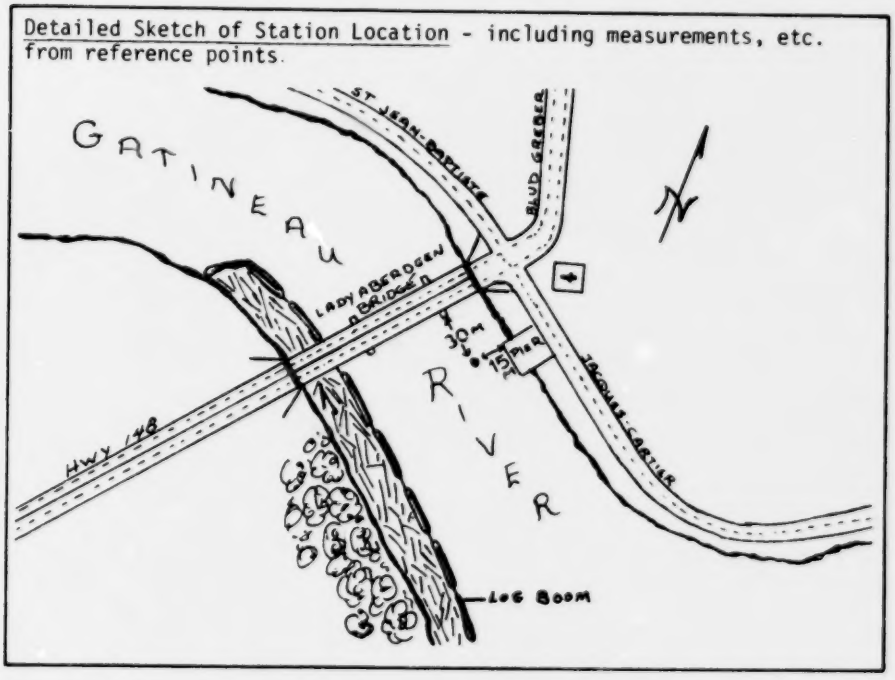


Figure 6. Station location description forms and maps (continued).

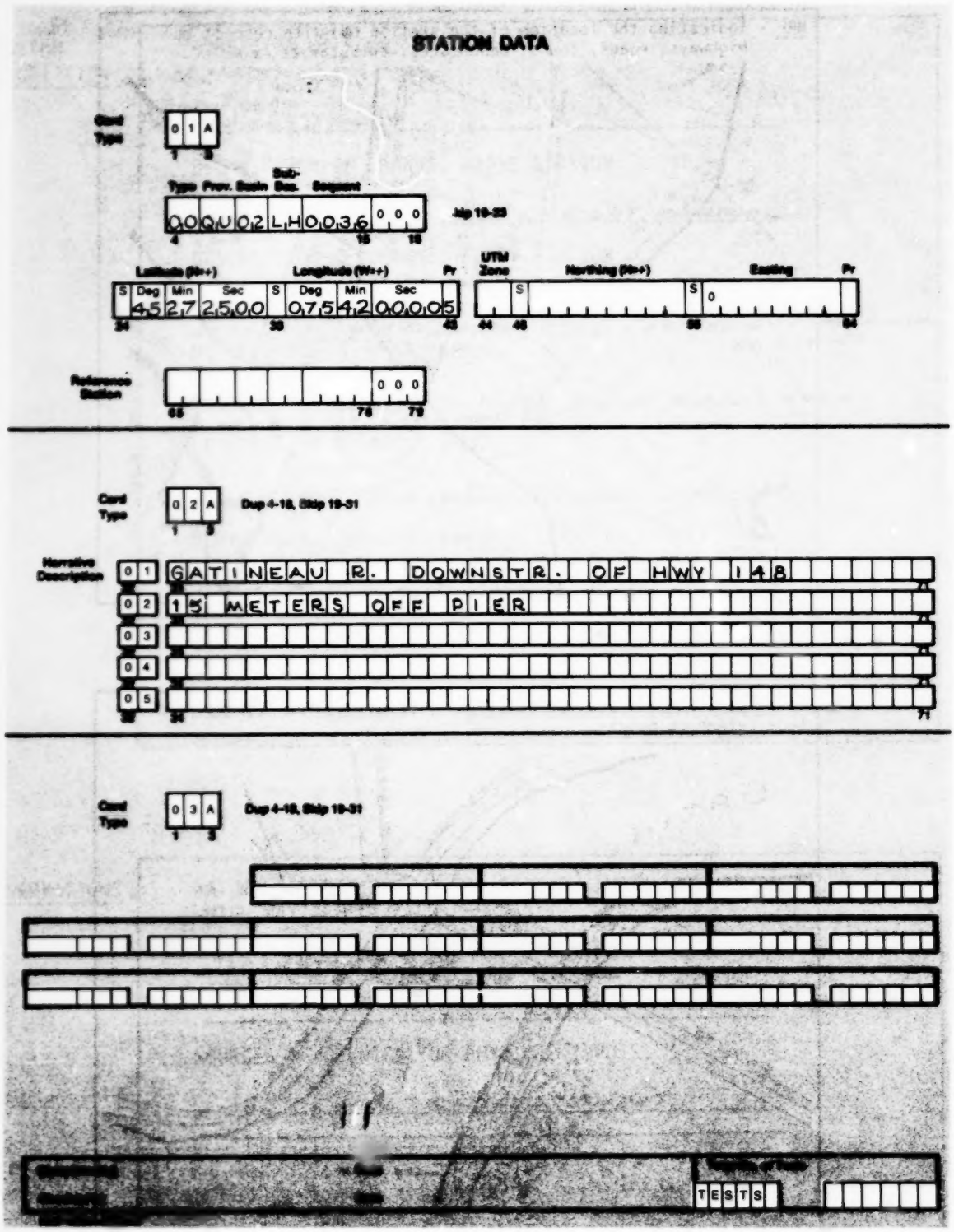

STATION DATA
Card Type
0 1 A
Type Prov. Basin Sub-Bas. Segment
Q Q Q U 0 2 L H 0 0 3 6 0 0 0
Skip 19-23
Latitude (N++) Longitude (W++) Pr
S Deg Min Sec S Deg Min Sec
4 5 2 7 2 5 0 0 0 7 5 4 2 0 0 0 0 5
UTM Zone Northing (N++) Easting Pr
S S 0
Reference Station
0 0 0
Card Type
0 2 A
Dup 4-18, Skip 19-31
Narrative Description
0 1 G A T I N E A U R. D O W N S T R. O F H W Y 1 4 8
0 2 1 5 M E T E R S O F F P I E R
0 3
0 4
0 5
Card Type
0 3 A
Dup 4-18, Skip 19-31
T E S T S

Figure 7. Station data form.

Data collected by provincial agencies may be stored on NAQUADAT by means of a different two-digit code to indicate the province:

Alberta	AT
British Columbia	CB
Manitoba	MN
New Brunswick	BR
Quebec	PQ
Saskatchewan	SK

Basin - a two-digit numerical code ranging from 01 to 10.

Subbasin - a two-digit alphabetical code.

The basin and subbasin codes are obtained from maps available in the Water Quality Branch. These maps were obtained from the Water Survey of Canada (Water Resources Branch, 1977), with the basin and subbasin codes used by the two branches being identical. A map of Canada showing these codes can be found in the pocket at the back of the *NAQUADAT Users Manual* (Demayo and Hunt, 1975).

Sequential Number - a four-digit numerical code starting with 0001. This number is assigned by the regional office.

2. Geographical Coordinates - Latitude and longitude or UTM (Universal Transverse Mercator) coordinates

This field is self-explanatory. Only one of the two types of coordinates need be given when adding a station to the NAQUADAT file. In NAQUADAT, all geographical coordinates are stored as latitude and longitude, with minutes and seconds given as a fraction of a degree. Thus, if the initial information has been given in UTM units, the system automatically converts it to latitude and longitude before storing it. Every subsequent retrieval will print the location in latitude and longitude.

The latitude and longitude values are obtained from 1:50 000 or 1:250 000 (for those areas that have not been mapped to 1:50 000 scale) topographical maps. If necessary and if available, navigational charts can be used to provide more accurate latitude and longitude values than the topographical maps. When a water quality sampling point coincides with a Water Survey of Canada station, the geographical coordinates can be obtained directly from the "Surface Water Data Reference Index" (Water Resources Branch, 1979).

The Latitude and Longitude Fields consist of the following subfields:

(a) S (one digit): sign, always blank.

(b) DEG (two and three digits, respectively): the degrees latitude or longitude.

(c) MIN (two digits): the minutes latitude or longitude.

(d) SEC (four digits): the seconds in the first two digits and the fraction of a second (decimal) in the last two digits. If this fraction is not available, then two "0" (zeros) must always be entered after the implied decimal point.

(e) PR (one digit): precision used to indicate accuracy of the latitude and longitude coordinates. For instance, if the location was determined to the nearest 10 s, then a 5 would be entered in the PR column. If the locations were determined on a 1:250 000 or 1:500 000 scale map, the location would probably be determined to the nearest minutes, and therefore a PR equal to 7 would be used.

Table 5 shows various formats for latitude and longitude values with the corresponding precision codes.

Table 5. Formats and Precision Codes for Sampling Locations

Latitude and Longitude*					
DEG†	MIN	SEC	Rounding off to the nearest:	Code	Radius (r)‡
XXX	XX	XX.XX	±0.03 sec or better	0	$r \leq 1$ m
XXX	XX	XX.X0	±0.1 sec	1	1 m < $r \leq$ 3 m
XXX	XX	XX.X0	±0.3 sec	2	3 m < $r \leq$ 10 m
XXX	XX	XX.00	±1 sec	3	10 m < $r \leq$ 30 m
XXX	XX	XX.00	±3 sec	4	30 m < $r \leq$ 100 m
XXX	XX	XX.00	±10 sec	5	100 m < $r \leq$ 300 m
XXX	XX	XX.00	±30 sec	6	300 m < $r \leq$ 1 km
XXX	XX	00.00	±1 min	7	1 km < $r \leq$ 3 km
XXX	XX	00.00	±5 min	8	3 km < $r \leq$ 10 km
XXX	XX	00.00	±10 min or worse	9	$r \leq$ 10 km

* X represents an actual number (0-9) and 0 is a zero.

† The DEG subfield for latitude has only two digits; DEG subfield for longitude must have leading zero if < 100.

‡ Radius, in metres, from the specified location within which the sample was taken.

Source: *NAQUADAT Users Manual* (Demayo and Hunt, 1975).

The UTM (Universal Transverse Mercator Grid) Coordinates Field consists of the following subfields:

(a) UTM Zone: given on topographical maps.

(b) S: sign, always blank.

(c) Northing and Easting: obtained from topographical maps. These values must be entered with one implied decimal position.

(d) PR: precision, obtained from Table 5.

3. Narrative Description

This section contains a narrative description of the sampling location. This description is free format and it supplements the geographical coordinates in specifying the sampling location. NAQUADAT normally permits up to 5 lines of 38 characters each to be used for the "Narrative Description." Although this can be extended, most reports are printed with only two lines of description; however, it is advisable to format the first two lines as a self-contained heading.

Generally, the narrative description begins with the name of the river, stream, lake or reservoir followed by its location (upstream, downstream, etc.) and its distance (to 0.1 km or better) from the nearest town, city, important bridges, highways, or other important fixed landmarks. The name of the province or territory always ends the narrative description.

It is recommended that only dictionary-approved abbreviations be used and that local names for landmarks be avoided.

If water is sampled at a Water Survey of Canada location, reference should be made to the latest edition of the "Surface Water Data Reference Index" (Water Resources Branch, 1979) to obtain the geographical coordinates, basin codes, and a narrative description. Any information obtained from the Reference Index should be checked and completed where it does not fulfill the needs of NAQUADAT.

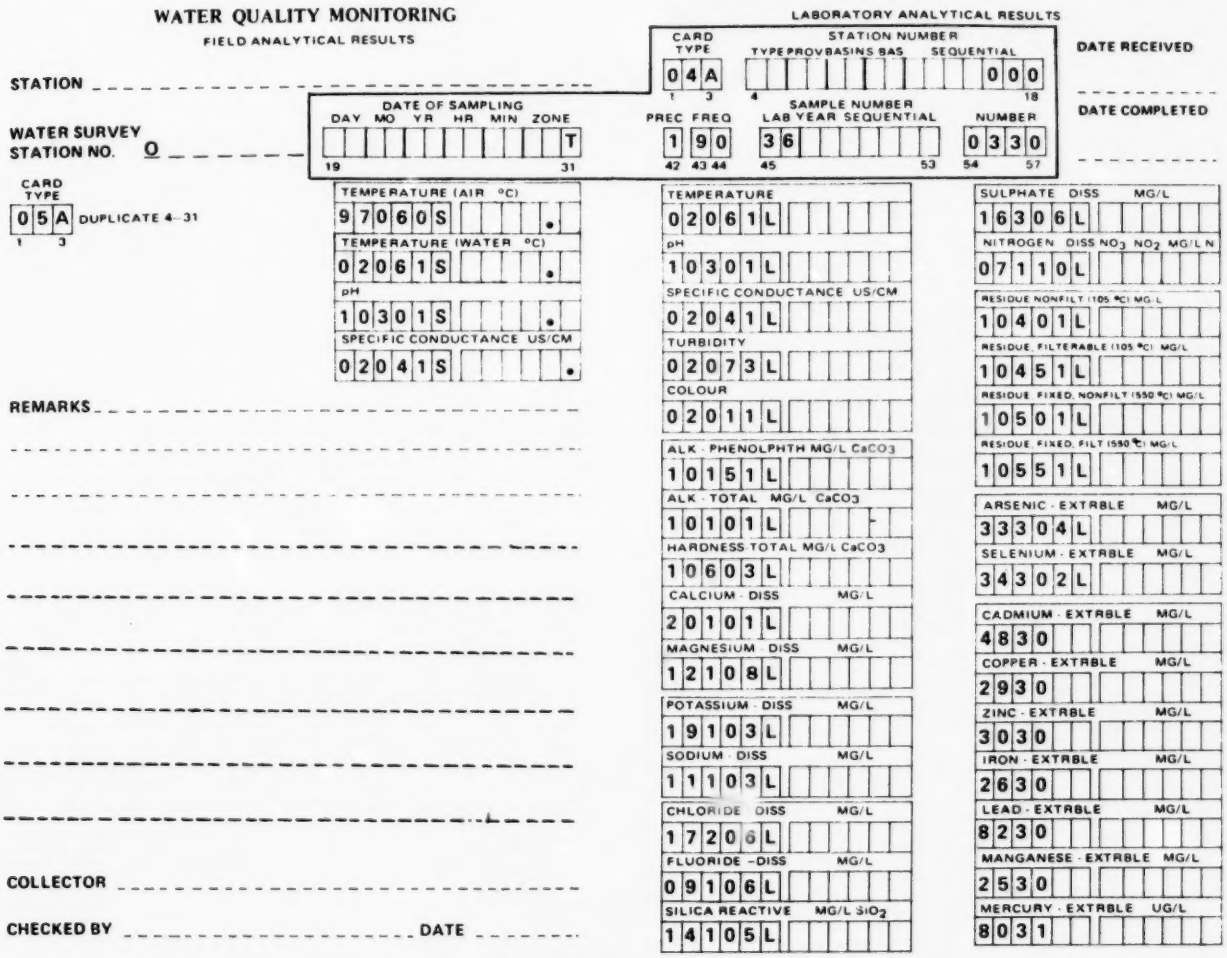

WATER QUALITY MONITORING
FIELD ANALYTICAL RESULTS
LABORATORY ANALYTICAL RESULTS
STATION
WATER SURVEY STATION NO. O
CARD TYPE
DATE OF SAMPLING DAY MO YR HR MIN ZONE
CARD TYPE STATION NUMBER TYPE PROV BASINS BAS SEQUENTIAL
SAMPLE NUMBER PREC FREQ LAB YEAR SEQUENTIAL NUMBER
DATE RECEIVED
DATE COMPLETED
05A DUPLICATE 4-31
TEMPERATURE (AIR °C) 97060S
TEMPERATURE (WATER °C) 02061S
pH 10301S
SPECIFIC CONDUCTANCE US/CM 02041S
TEMPERATURE 02061L
pH 10301L
SPECIFIC CONDUCTANCE US/CM 02041L
TURBIDITY 02073L
COLOUR 02011L
ALK - PHENOLPHTH MG/L CaCO3 10151L
ALK - TOTAL MG/L CaCO3 10101L
HARDNESS TOTAL MG/L CaCO3 10603L
CALCIUM - DISS MG/L 20101L
MAGNESIUM - DISS MG/L 12108L
POTASSIUM - DISS MG/L 19103L
SODIUM - DISS MG/L 11103L
CHLORIDE DISS MG/L 17206L
FLUORIDE -DISS MG/L 09106L
SILICA REACTIVE MG/L SiO2 14105L
SULPHATE DISS MG/L 16306L
NITROGEN DISS NO3 NO2 MG/L N 07110L
RESIDUE NONFILT (105 °C) MG/L 10401L
RESIDUE, FILTERABLE (105 °C) MG/L 10451L
RESIDUE, FIXED, NONFILT (550 °C) MG/L 10501L
RESIDUE, FIXED, FILT (550 °C) MG/L 10551L
ARSENIC - EXTRBLE MG/L 33304L
SELENIUM - EXTRBLE MG/L 34302L
CADMIUM - EXTRBLE MG/L 4830
COPPER - EXTRBLE MG/L 2930
ZINC - EXTRBLE MG/L 3030
IRON - EXTRBLE MG/L 2630
LEAD - EXTRBLE MG/L 8230
MANGANESE - EXTRBLE MG/L 2530
MERCURY - EXTRBLE UG/L 8031
REMARKS
COLLECTOR
CHECKED BY
DATE

Figure 8. Field sheet for use with NAQUADAT or similar computer systems.

4. Reference Station

Often it is necessary to indicate the distance of a sampling location from a given point. NAQUADAT uses the "Reference Station" for this purpose. The chosen reference point, which could be, for example, the mouth of a river or the confluence point of two rivers, is given a "Station Number" exactly in the same manner as a number is assigned to the sampling location itself (see above). This number is entered in the "Reference Station" field on the station card shown in Figure 7. The distance of the sampling location from this reference point is then given as a station parameter [see (5) below].

Besides providing a more precise identification of a station location, the distance from a reference point can be used for obtaining "concentrations vs. distance" type plots often used in water quality data interpretation.

5. Station Parameters

This section, (03) on the station card (Fig. 7), contains additional information about the sampling location. The information is entered via a six-digit parameter code number indicating the type of information and a value of up to six digits.

The following parameter code numbers are considered to be "Station Parameters" and are stored with the station information:

- 97002F - average depth of water at sampling station, in metres.
- 97011F - distance downstream from reference station, in kilometres.
- 97900S - Water Survey of Canada (WSC) corresponding station number. Because the basin and subbasin codes used by NAQUADAT and by Water Survey of Canada are identical, only the sequential number, i.e., the last three numeric digits, of the WSC number is given.

5.2 Field Sheets

Perhaps one of the most important steps in a sampling program is the recording of observations, sampling date, time and location on the field sheets. All field measurements and records must be completed before a station is left.

The interpretation of water quality data cannot be accomplished without all supporting information. Observations of weather, dead fish, algal growth, slicks on the water surface and other phenomena may make a significant difference in explaining anomalies in data. Do not hesitate to record all observations, no matter how trivial they may seem.

Two examples of a systematic format for recording field analyses and observations are provided in Figures 8 and 9. The format shown in Figure 8 is appropriate for those groups using the NAQUADAT or similar computer systems for storing their results. The format of Figure 9 can be used by any group doing water quality work. Both formats can be adapted to fit a particular situation specific to a particular agency.

5.2.1 *Completion of Field Sheets*

Station Number:
NAQUADAT Number, e.g. 00QU02LH0036 (or other identification code), is entered here to identify a sampling location. This number can be obtained from Station Location Description.

Description:
Each sampling site must be identified by a brief narrative explanation of the water body sampled indicating the sources and location, e.g. Gatineau River near Pointe-Gatineau.

Date of Sampling:
Enter the day, month, year.

Time of Sampling:
Enter the time of sample collection (according to the 24-h clock), the time zone and whether standard or daylight time, e.g. HR 15 MIN 45 TIME ZONE EDT.

Sampled by:
Enter name of field investigator.

5.2.1.1 Field Measurement Parameters

Values should be entered next to the appropriate NAQUADAT parameter code (Water Quality Branch, 1982).

Water Temperature:
Enter temperature of the water sampled in degrees Celsius.

Air Temperature:
Enter temperature of the air at time of sampling in degrees Celsius.

pH:
Enter the *in situ** or field pH value of the water sampled.

Specific Conductance:
Enter the *in situ* or field conductivity and state whether it was adjusted to 25°C.

Dissolved Oxygen:
Enter the *in situ* or field measurements of dissolved oxygen concentration.

Turbidity:
Enter turbidity of water as obtained from a calibrated instrument. General observations on turbidity can be entered at the end of this section under "Remarks."

Depth of Water:
Enter depth from surface.

---

* *In situ* measurements are made directly in the water body being sampled. Field measurements are made on shore on the water sample just collected.

STATION NO. _____

DESCRIPTION: _____

_____

DATE OF SAMPLING DY _____ MO _____ YR _____

TIME OF SAMPLING HR _____ MI _____ TIME ZONE _____

SAMPLED BY _____

FIELD MEASURED PARAMETERS

Water Temp. °C _____ Air Temp. °C _____

pH _____ Specific Cond. _____ Diss. Oxygen _____ Turb. _____

Depth of Water _____ Depth at which Sample Taken _____

Ice Thickness _____

Other _____

Remarks _____

_____

INSTRUMENT CALIBRATION

Diss. Oxygen Meter Model _____ Winkler Calibration _____ mg/L

Meter Reading before Adjustment _____

Conductivity Meter Model _____

pH Meter Model _____ Calibration Buffers Used _____

Remarks _____

_____

WATER QUANTITY MEASUREMENT DATA

Location Description _____

_____

Description of Gauge _____

_____

Stage Height _____

Time _____

Figure 9. General format for a field sampling sheet.

SAMPLING APPARATUS USED AND PROCEDURES

SAMPLE SPECIFICS

	Container Material	Vol. Collected	Preservation	Quality Control
Major Ions				
Metals				
Organics				
Pesticides & Herbicides				
Mercury				
Phenols				
Nutrients				
BOD & COD				
Others				

QUALITY CONTROL REMARKS

GENERAL REMARKS

MODE OF TRANSPORT

SHIPPING DATE

Figure 9. General format for a field sampling sheet (continued).

Depth at Which Sample Taken:
Enter water depth.

Ice Thickness:
Enter ice thickness.

Other:
Enter any other pertinent observations, e.g., estimate of the direction and velocity of the wind, percent cloud cover, snow depth, etc.

5.2.1.2 Instrument Calibration

Dissolved Oxygen Meter Model:
Enter make and model number of dissolved oxygen meter.

Winkler Calibration:
Enter dissolved oxygen concentration measured in milligrams per litre (to be used to calibrate dissolved oxygen meter).

Meter Reading before Adjustment:
Enter meter reading prior to adjustment based on Winkler calibration in milligrams per litre.

Conductivity Meter Model:
Enter make and model number of meter used.

pH Meter Model:
Enter make and model number of meter used.

Calibration Buffers Used:
Enter pH range for which meter is calibrated.

Remarks:
Record any problems such as erratic needle movement or continual slow drift.

Water Quantity Measurement Data:
Record the description of the gauge and location along with the stage height and time.

Sampling Apparatus Used and Procedures:
Enter type of water sampler used (e.g. sampling iron, metal Kemmerer), and type of sample taken (e.g. grab-point or depth-integrated samples).

Sample Specifics:
Record for each water quality parameter or group of parameters the bottle used, volume of water collected, means of preservation, and whether or not a sample was collected for quality control purposes. Consult Chapter 6 for recommended bottles, sample sizes, and preservation techniques.

Quality Control Remarks:
Record the specifics of the quality control requirements, i.e., number of duplicates, acid blanks, time of collection.

General Remarks:
Enter any general field observations which may affect water quality. These observations may include unusual colour or odour of the water, excessive algal growth, oil slicks, surface films, or a heavy fish kill. Observations recorded in this section may prompt the field investigator to take additional "observation-based" samples (Section 2.2).

Field Filtration and Preservation Procedures

In an aquatic environment, inorganic and organic substances can be found in a variety of forms, such as free or complexed; dissolved, particulate or sorbed onto suspended sediments and biomass; and associated with the bottom materials.

Although the question has been addressed in the scientific literature, so far no clear consensus has emerged on which chemical-physical species of a substance should be measured when monitoring water quality. This depends on the particular system being studied, on the purpose of the study and the biological availability of the various species of the substances being studied. The issue of biological availability is also far from being resolved. For many substances the biological availability is directly proportional to their concentration in dissolved phase. Also in the case of most metals, for example, typically the concentration in the dissolved phase is low compared with the suspended or colloidal particles. However, the greatest quantity of each element, sometimes over 90%, occurs in the dissolved phase because the dissolved solids comprise a large percentage of total solids. For these reasons in water quality work it is very important to have the capability of measuring the "dissolved" constituents. The definition used by the Water Quality Branch for "dissolved" phase is that which passes through a 0.45-μm membrane filter. Optimally, the filtration should be carried out in the field during or immediately after sample collection and must be followed by the appropriate preservation.

6.1 Filtration

To determine the concentration of dissolved inorganic constituents (e.g. metals and anions), it is necessary to filter the sample through a 0.45-μm membrane filter immediately after collection. The filtrate for metals analysis should be preserved as outlined in Table 6; the filtrate for anion analysis is not preserved. The volume of sample needed is indicated by laboratory personnel. The filter and filtration apparatus require laboratory pre-treatment and should be rinsed with a portion of the collected sample before the filtrate is collected.

Samples requiring analysis for organic constituents are filtered immediately after collection using a glass-fibre filter. After filtration the filtrate may be analyzed for dissolved organic constituents, and the filter supporting the particulate fraction is available for particulate organic analysis.

The filtration procedure requires maintaining a vacuum in the filtration apparatus; either an electrical or manual pump must be used. If an electrical type is employed filtration will require access to electrical services or the operation of a mobile power unit.

6.1.1 *General Apparatus Washing Procedures*

A. Before or after each field trip:

(1) Wash and scrub filtration units with non-phosphate detergent.

(2) Rinse with tap water and soak overnight in 5% HCl.

(3) Clean inside (only) of tubing by soaking for 16 h or more with 5% HCl.

(4) Rinse unit and tubing with tap water followed by distilled or deionized water.

B. Before each filtration:

(1) Rinse unit and tubing with distilled or deionized water.

(2) Continue with procedures as outlined in Section 6.1.2.

C. After last filtration of the day:

(1) Rinse unit with 5% HCl (pump through the tubing).

(2) Scrub as necessary.

(3) Rinse with tap water followed by distilled or deionized water.

Note: If at any time the unit appears to contain residue from filtration which has not been removed by procedures outlined in B and C, proceed to A.

6.1.2 *Filtration Procedures*

6.1.2.1 Particulate Carbon and Nitrogen

Immediately following the collection of the required sample:

(a) Using a pair of tweezers, place a pre-treated* glass-fibre filter paper on the filtration holder, taking care not to touch the paper with fingers.

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* Clean the glass-fibre filters by placing them in a preheated muffle furnace (450°C) for 1 h to remove organic mater. Do not use filters that are bent. Store filters in a sealed dry container.

Place the filtration bowl on the filter and clamp or twist securely.

Note: Both the filtration bowl and vacuum flask must be acid washed and stored in heat-sealed polyethylene bags. Each filtration unit may only be used once between acid washes.

(b) Filter a measured volume of the well-shaken water sample. Record the volume of water filtered.

(c) After the water sample has passed through the filter, wash the inside of the filter bowl with a few millilitres of carbon- and nitrogen-free distilled water to ensure that all particulate material is on the filter.

(d) Acid wash to remove carbonates by carefully adding 2-5 mL 0.1 *N* sulphuric acid (0.3% v/v sulphuric acid) to the filter. Continue filtering.

(e) Rinse the filter with 2-5 mL carbon- and nitrogen-free distilled water.

(f) Remove the filtration bowl and continue filtering until all excess water has been removed from the filter.

(g) Transfer the filter to a labelled petri dish. Store the petri dishes at 4°C.

(h) Periodically, distilled water blank filters should be prepared using the filtration steps above to monitor the background concentration levels.

6.1.2.2 Chlorophyll *a*

(a) Samples for chlorophyll *a* should be filtered immediately.

(b) Preserve the sample with 0.2 mL of $MgCO_3$ solution (1.0 g $MgCO_3$ in 100 mL H_2O) just as the last of the sample is being filtered.

(c) Mount a clean filter head (Millipore Pyrex filtering head) and a GF/C filter (Whatman) or a Millipore filter of suitable pore size.

(d) Mix the sample well in a 500-mL polyethylene bottle; exposure to light must be avoided.

(e) As much sample as possible is passed through the glass filter paper and volume recorded.

(f) Non-turbid samples will generally pass through the filter completely (approximately 450 mL). Highly turbid samples may only pass 50-100 mL.

(g) The GF/C filter should not be permitted to dry between filtering sequences.

(h) After the sample is filtered, the sides of the filter head should be rinsed with approximately 50 mL of demineralized water.

(i) The GF/C filter is then permitted to dry 5 s after the rinse has passed.

(j) Fold the GF/C filter in half using two pairs of forceps, place it in a labelled petri dish, and then place the petri dish in a sub-zero freezer unit.

(k) Record the total volume of filtered samples on labelled petri dish.

6.2 Preservation Techniques

Between the time that a sample is collected in the field and until it is actually analyzed in the laboratory, physical changes, and chemical and biochemical reactions may take place in the sample container which will change the intrinsic quality of the water sample. It is necessary, therefore, to preserve the samples before shipping to prevent or minimize these changes. This is done by various procedures such as keeping the samples in the dark, adding chemical preservatives, lowering the temperature to retard reactions, freezing samples, extraction procedures, field column chromatography, or by a combination of these methods.

The preservation methods recommended by the Water Quality Branch are discussed briefly here and summarized in Tables 6 and 7.

6.2.1 *Chemical Addition*

This method, which includes acidification, is used for preserving the water samples for a variety of tests, including most dissolved metals and phenoxy acid herbicides. Care must be taken in using only "reagent" grade chemicals such that the water sample is not contaminated by impurities in the added preservatives. Some samples for biological analysis also require chemical preservation.

6.2.2 *Freezing*

Freezing may be acceptable for certain analyses but is not used as a general preservation technique because it can cause physico-chemical changes, e.g. formation of precipitates and loss of dissolved gases, which might affect the sample composition. Also, solid components of the sample change with freezing and thawing, and a return to equilibrium followed by high-speed homogenization may be necessary before any analysis can be run.

6.2.3 *Refrigeration*

Refrigeration at 4°C is a common preservation technique which is widely used in fieldwork. However, it does not maintain the complete integrity of all constituents. In some cases it may affect the solubility of some constituents and cause them to precipitate. Refrigeration is often used in conjunction with chemical addition (Table 6).

6.2.4 *Practical Aspects of Preservation*

An important practical aspect of preservation is a consistent routine to ensure that all samples requiring preservation receive the immediate treatment they need. This is particularly important when a chemical preservative is added, since these additions may not produce an easily detectable change in sample appearance. It may be advisable to mark or flag each preserved sample to

Table 6. Sample Containers and Preservation for Constituents in Water

Parameter	Recommended container*	Preservative	Maximum permissible storage time
Acidity	Polyethylene	Cool, 4°C	24 h
Alkalinity	Polyethylene	Cool, 4°C	24 h
Aluminum	Polyethylene	2 mL Conc. HNO_3/L sample	6 months
Antimony	Polyethylene	2 mL Conc. HNO_3/L sample	6 months
Arsenic	Polyethylene	Cool, 4°C	6 months
Barium	Polyethylene	2 mL Conc. HNO_3/L sample	6 months
Beryllium	Polyethylene	2 mL Conc. HNO_3/L sample	6 months
BOD	Polyethylene	Cool, 4°C	4 h
Boron	Polyethylene	Cool, 4°C	6 months
Cadmium	Polyethylene	2 mL Conc. HNO_3/L sample	6 months
Calcium	Polyethylene	Cool, 4°C	7 days
Carbamate pesticides†	Glass	H_2SO_4 to pH <4, 10 g Na_2SO_4/L sample	Preferable to extract immediately
Carbon, inorganic	Polyethylene	Cool, 4°C	24 h
Carbon, organic‡	Polyethylene	Cool, 4°C	24 h
Carbon, particulate	Plastic petri dish	Filter using GF/C filter; cool, 4°C	6 months
Chloride	Polyethylene	Cool, 4°C	7 days
Chlorinated hydrocarbon pesticides†	Glass	Cool, 4°C	Preferable to extract immediately
Chlorophyll	Plastic petri dish	Filter on GF/C filter; freeze -20°C	7 days
Cholesterol	Glass	1 mL Conc. H_2SO_4/L sample	24 h
Chromium	Polyethylene	2 mL Conc. HNO_3/L sample	6 months
Cobalt	Polyethylene	2 mL Conc. HNO_3/L sample	6 months
COD	Polyethylene	Cool, 4°C	24 h
Colour	Polyethylene	Cool, 4°C	24 h
Copper	Polyethylene	2 mL Conc. HNO_3/L sample	6 months
Coprostanol	Glass	1 mL Conc. H_2SO_4/L sample	24 h
Cyanide	Polyethylene	1 mL 10% NaOH/100 mL sample	24 h
Dissolved oxygen Winkler titration	Glass	Fix on site	6 h
Fluoride	Polyethylene	Cool, 4°C	7 days
Hardness	Polyethylene	Cool, 4°C	24 h
Iodide	Polyethylene	Cool, 4°C	24 h
Iron	Polyethylene	2 mL Conc. HNO_3/L sample	6 months
Lead	Polyethylene	2 mL Conc. HNO_3/L sample	6 months
Lithium	Polyethylene	2 mL Conc. HNO_3/L sample	6 months

* Teflon containers can also be used to replace either the polyethylene or the glass containers shown in the table.

† In some WQB regions, depending upon the specific compound to be analyzed within these groups and the method of analysis employed, certain organic solvents are also added to samples in the field in measured amounts. Preliminary research results have shown that this might be a better method of preservation than only cooling to 4°C.

‡ In some WQB regions these parameters are measured from the same bottle. Therefore the preservation method is the same, i.e. 2 mL 30% v/v H_2SO_4/L sample + cool 4°C. This method of preservation for organic carbon and total phosphorous has not been corroborated by any WQB studies.

§ If the whole sample is autoclaved in the original sample container.

Note: This table has been adapted from the *Analytical Methods Manual* (Water Quality Branch, 1981).

Table 6. Continued

Parameter	Recommended container	Preservative	Maximum permissible storage time
Magnesium	Polyethylene	Cool, 4°C	7 days
Manganese	Polyethylene	2 mL Conc. HNO_3/L sample	6 months
Mercury	Glass or Teflon	1 mL Conc. H_2SO_4, 1 mL 5% $K_2Cr_2O_7$ solution/100 mL sample	1 month
Molybdenum	Polyethylene	2 mL Conc. HNO_3/L sample	6 months
Nickel	Polyethylene	2 mL Conc. HNO_3/L sample	6 months
Nitrogen			
Ammonia[‡]	Polyethylene	Cool, 4°C, 2 mL 40% H_2SO_4/L	24 h
Kjeldahl	Polyethylene	Cool, 4°C	24 h
Nitrate + nitrite[‡]	Polyethylene	Cool, 4°C	24 h
Organic nitrogen	Polyethylene	Cool, 4°C	24 h
Organic-particulate	Plastic petri dish	Filter using GF/C filter; cool, 4°C	6 months
NTA			
Polarographic	Polyethylene	Treated with 0.5% of 37% solution of formaldehyde; filter using 0.45-μm membrane filter	24 h
GLC	Glass	5 mL Conc. HCl/L sample	24 h
Oil and grease	Glass	Cool, 4°C, 5 mL 1 + 1 H_2SO_4/L (H_2SO_4 to pH <2)	24 h
Organophosphorus pesticides[†]	Glass	Cool, 4°C, 10% HCl to pH 4.4	No holding; extraction on site preferable
Pentachlorophenol	Glass	H_2SO_4 to pH <4, 0.5 g $CuSO_4$/L; cool, 4°C	24 h
pH	Polyethylene	None	6 h
Phenolics	Glass	H_3PO_4 to pH <4, 1.0 g $CuSO_4$/L; cool, 4°C	24 h
Phenoxy acid herbicides	Glass	H_2SO_4 to pH <2; cool, 4°C	Preferable to extract immediately
Phosphorus			
Dissolved	Glass	Filter using 0.45-μm membrane filter on site	24 h
Inorganic	Glass	Cool, 4°C	24 h
Ortho	Glass	Cool, 4°C	24 h
Total[‡]	Glass	Cool, 4°C	1 month[§]
Potassium	Polyethylene	Cool, 4°C	7 days
Residue	Polyethylene	Cool, 4°C	7 days
Selenium	Polyethylene	Cool, 4°C	6 months
Silica	Polyethylene	Cool, 4°C	7 days
Silver	Polyethylene	0.4 g Disodium EDTA/100 mL sample	10 days
Sodium	Polyethylene	Cool, 4°C	7 days
Specific conductance	Polyethylene	Cool, 4°C	24 h
Strontium	Polyethylene	2 mL Conc. HNO_3/L sample	6 months
Sugar	Glass	Cool, 4°C	24 h
Sulphate	Polyethylene	Cool, 4°C	7 days
Turbidity	Polyethylene	Cool, 4°C	7 days
Vanadium	Polyethylene	2 mL Conc. HNO_3/L sample	6 months
Zinc	Polyethylene	2 mL Conc. HNO_3/L sample	6 months

Table 7. Sample Containers and Preservation for Constituents in Sediment

Parameter	Recommended container	Preservative	Maximum permissible storage time
Carbon	Polyethylene or polypropylene bag or jar	Freeze, -20°C	6 months
Chlorinated hydrocarbon pesticides	Glass jar or aluminum cannister	Freeze, -20°C	Extract as soon as possible
Mercury	Polyethylene or polypropylene bag or jar	Freeze, -20°C	6 months
Metals	Polyethylene or polypropylene bag or jar	Freeze, -20°C	6 months
Nitrogen	Polyethylene or polypropylene bag or jar	Freeze, -20°C	6 months
Oil and grease	Glass jar or aluminum cannister	Freeze, -20°C	Extract as soon as possible
Phosphorus	Polyethylene or polypropylene bag or jar	Freeze, -20°C	6 months

Note: This table has been adapted from the *Analytical Methods Manual* (Water Quality Branch, 1981).

ensure that no sample is forgotten or treated more than once.

Safe and accurate field addition of chemical preservatives also requires special precautions. Pre-calibrated Repipets™ and other automatic pipettes now ensure accurate field addition as well as eliminating the safety hazard of pipetting acids by mouth. It is necessary when using automatic pipettes to check that air bubbles are absent from the delivery tube. Automatic dispensers must be primed so that subsequent samples will receive the correct aliquot of preservative. It is important that each pipette be unique to a preservative so that there is no possibility of cross-contamination from one preservative to another. Finally, it is advisable to label clearly all preservative bottles used in the field with their contents and the volume to be used, for example, conc. nitric acid, add 2 mL/L of sample.

Sampling for Radioactivity Measurements

7.1 Sources of Radioactivity in Water

Radioactivity in water may be of natural or man-made origin. The main natural processes that introduce radioactivity into water are the weathering of rocks containing radioactive minerals and fallout of cosmic-ray-produced nuclides. The major sources of artificial radioactivity are uranium mining and the nuclear power industries, nuclear weapons testing, and the peaceful applications of nuclear materials and devices.

The principal radionuclides introduced naturally into surface and groundwaters are uranium, radium-226, radium-228, radon, potassium-40, tritium and carbon-14. All but the last two derive from radioactive minerals. In areas where radioactive minerals are abundant, natural uranium is the major radioactive constituent present in water. Tritium and carbon-14 are produced by the interaction of cosmic-ray neutrons with nitrogen in the upper atmosphere. The tritium is eventually rained out as tritiated water, and the radiocarbon is incorporated into atmospheric carbon dioxide. Both radionuclides are also produced by thermonuclear weapons testing. Tritium is also a fission product, and since 1970, the nuclear power industry has probably been the largest source of tritium.

Strontium-90, cesium-135 and plutonium-239 are the major radioisotopes of concern in man-made radioactivity in water.

Dissolved and particulate radioactivity in water is controlled by the same mechanisms that affect other trace and macro constituents in the geohydrologic environment. The geochemical behaviour of a daughter element may be grossly different from that of the radioactive parent, although its occurrence, distribution and transport may be governed by the parent.

7.2 Collection and Preservation of Samples

Samples are collected in 4-L polyethylene bottles, acidified with 8-mL of concentrated HCl, and then shipped to the laboratory.

Compositing is the procedure used in the Water Quality Branch to sample for radioactivity measurements monthly and then to analyze the annual composite samples. These are made up by mixing, in a separate bottle, 400-mL aliquots from each monthly sample. If a significant level of radioactivity over the background levels is found, then the samples making up the composite are analyzed individually to locate the sample(s) which has the higher than expected radioactivity level.

Sampling for Microbiological Analysis

It is very important that all water samples submitted for microbiological analysis be collected as aseptically as possible in order to reflect accurately microbiological conditions at the time of sample collection.

The essential information for sampling for microbiological analysis is given here. More detailed information about the various aspects of microbiological sampling and analysis can be found in the *Methods of Microbiological Analysis of Waters, Wastewaters and Sediments* (National Water Research Institute, 1978).

8.1 Sample Containers

Microbiological samples are usually collected in sterile 200-mL or 500-mL wide-mouthed glass or nontoxic plastic bottles with cork or screw caps. The capped or stoppered bottle mouth should be covered with sterile heavy-duty paper or with aluminum foil secured with string or an elastic band.

8.2 Sample Preservation

Whenever possible, water samples should be analyzed immediately after collection. If immediate processing is impossible, samples should be stored in the dark in melting ice. Storage under these conditions minimizes multiplication and die-off problems up to 30 h after collection. Samples should never be frozen.

8.3 Sampling Procedure

(a) Remove as one unit, the protective paper and stopper of the sample bottle.

(b) Do not rinse the sterile bottle.

(c) If the sample is to be collected in a hand-held fashion, hold the sample bottle near the base and plunge the bottle neck downward below the water surface 25-40 cm. Tilt bottle so that the neck points slightly upward and during filling push bottle horizontally forward in a direction away from the hand to avoid contamination. If a current is present, direct the mouth of the bottle against the current.

(d) If a bacteria sampling apparatus is used, fit the bottle into the spring clamp with the bottle mouth downward. The mechanism is designed so that the bottle is then rotated to the upright position under the water surface by the mechanical sampler.

(e) Remove bottle and decant sufficient water to leave a space of 3-4 cm between stopper and water line.

(f) Replace cap or stopper and paper covering, fasten cover and label bottle (if not pre-labelled) with a waterproof marking pencil.

(g) Place bottle in an ice chest.

(h) Record the time of sampling and depth of sample, water temperature and ambient temperature on the field sampling sheet. Description of the location can be obained from the station location description sheet.

Precipitation Sampling

The material contained in this chapter has been adapted from the *CANSAP MANUAL, the Canadian Network for Sampling Precipitation* (Atmospheric Research Directorate, 1980). In recent years it has become increasingly apparent that deposition of atmospheric pollutants to aquatic ecosystems is of major ecological significance. Most notable in this regard has been the demise of fish populations in various lakes of eastern Canada as a result of acidic precipitation, and the discovery of toxic organic contaminants in lakes far removed from anthropogenic sources.

A change in the chemical composition of precipitation is a good indicator of changes in atmospheric composition, since precipitation is an effective scavenging agent of many atmospheric substances, both solid and gaseous. Rainfall not only contributes hydrogen ions but also contributes significant amounts of other chemical constituents including trace metal nutrients and organic compounds.

This section discusses the criteria necessary for the collection of liquid and frozen precipitation samples.

9.1 Site Selection

Sampling sites should be selected to give accurate and representative information concerning the temporal and spatial variation of precipitation deposition of chemical constituents of interest. Important factors to take into consideration are prevalent wind trajectories, source areas for compounds of interest, frequency of precipitation events (rain, snow, hail, etc.), and other meteorological and atmospheric processes that influence the wet deposition.

Local criteria to consider are as follows:

(a) No moving sources of pollution, such as routine air, ground or water traffic should be within 100 m of the site.

(b) No surface storage of agricultural products, fuels or other foreign materials should be within 100 m of the site.

(c) No continuous sources of pollution should be within 50 km in the direction of the prevalent wind direction for the site and 30 km in all other directions.

(d) Sampler should be installed over undisturbed land, preferably grass-covered with no objects (including trees) within 5 m of the sampler.

(e) No object shall project onto the sampler with an angle greater than 30° from the horizontal to prevent splashing. Give particular attention to overhead wires.

It may not be possible to meet all of these criteria at individual sites. However, on completion of the relevant documentation as described in Section 5.1 of this manual, "Station Location Descriptions," the degree of departure from the preferred site requirements should be indicated.

9.2 Sample Collectors

Many types of collectors have been used to sample precipitation, from a basic plastic or stainless steel bucket placed on location at the beginning of a precipitation event, to a sophisticated sequential sampler designed to collect precipitation samples automatically at selected intervals during an event.

Most agencies in Canada are using the Sangamo Type A precipitation collector (Fig. 10). It is a "double bucket" collector; one bucket is used to collect precipitation only (wet deposition) and the other bucket is used to collect dust samples (dry deposition). The collector is equipped with an automatic sensing system which detects precipitation, liquid or frozen. At the onset of a precipitation event, the sensor detects the precipitation and the cover is moved from the "wet" bucket to the "dry" bucket. On cessation of the event, the cover is automatically returned to the "wet" bucket.

The sample container normally used with the sampler is a black polyethylene vessel, as shown in Figure 10. It consists of two black polyethylene parts. The top part is a removable rim which has been specially fabricated to ensure a sharply defined uniform area of collection. Both the rim and the bucket must be rinsed with distilled, deionized water each time a sample is removed. However, when sampling precipitation for organic contaminants a stainless steel bucket must be used.

9.3 Sample Collection Procedures

9.3.1 *Definition of the End of the Month*

For precipitation collection purposes only, the end of the month shall be defined as:

(a) 17:00 h G.M.T. on the last day of the current month or

(b) in the event that precipitation is occurring at the station at 17:00 G.M.T. on the last day of the month, the end of the month should be taken as the date and time that the precipitation ceases.

9.3.2 *Daily Sample Handling Procedure*

Any precipitation which has been collected over the preceding 24-h period must be removed from the collector and stored in the indoor storage container provided, using the procedures outlined below. If the station work

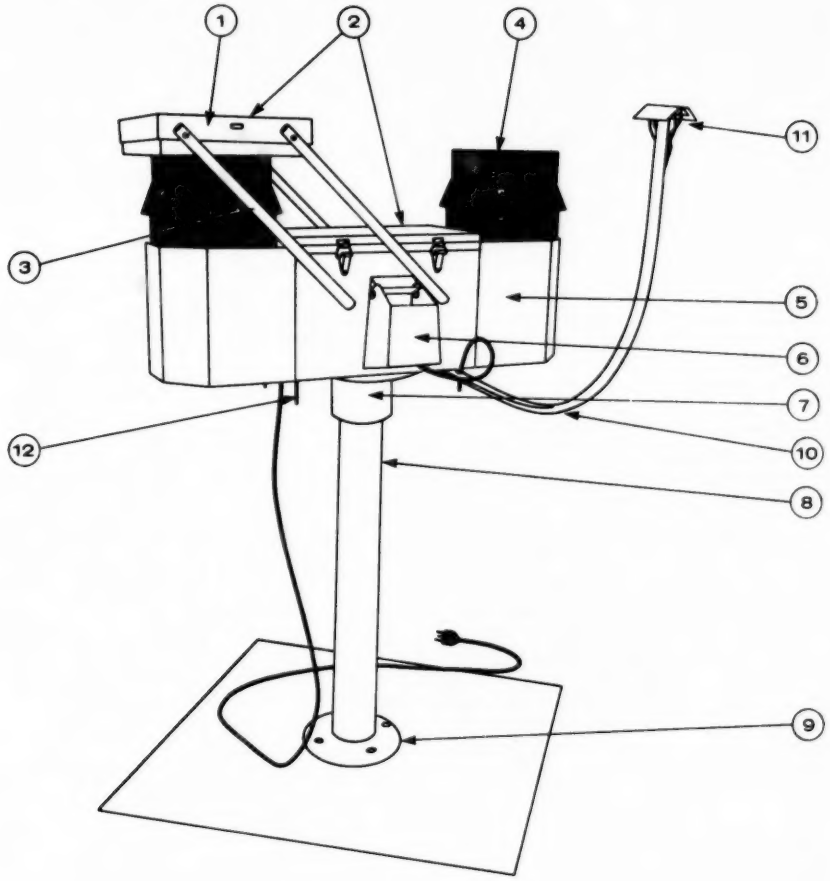


Figure 10. Sangamo Type A precipitation collector (Atmospheric Research Directorate, 1980):

1. Moveable cover
2. Splash screens
3. Cover support arms
4. Collection buckets
5. Housing
6. Cover plate
7. Mounting flange
8. Mounting pipe (7.5 cm I.D.)
9. Companion flange (18 cm)
10. Sensor head support arm
11. Sensor head
12. Housing vents and drain spouts

schedule does not permit a daily removal, e.g. weekends, or in the event that precipitation is occurring at the time the collector bucket should be checked, *do not remove* the daily sample but instead wait until the following day to remove the sample.

(a) Remove the vessel from the collector and carry it indoors.

(b) Remove the rim from the bucket.

Note: If sample is frozen (i.e. snow or ice) let it melt at room temperature before proceeding or place black bucket in pan of hot water to facilitate melting process.

(c) Using the graduated flask provided, measure the volume of the sample to the nearest 10 mL and note this figure on the Sample History Form together with the period over which the precipitation was collected, e.g. 25-07-81 to 26-07-81, 420 mL.

(d) Carefully pour the sample into an 8-L indoor storage container and re-cap the container immediately.

(e) Using the rinse bottle and deionized, distilled water, thoroughly rinse the bucket, rim and flask. Assemble the vessel and place it back in the collector. Place the plastic cover back on the graduated flask after rinsing it.

9.3.3 *Sample Handling Procedure at the End of the Month*

(a) Using the graduated flask, measure the total volume of sample accumulated over the month and note this on the Sample History Form in columns 30-33.

(b) Carefully pour the sample into a sample shipping bottle, filling it to within about 2 cm of the top if there is sufficient sample. Screw top onto shipping bottle securely.

(c) Discard any sample remaining in the graduated flask.

(d) Using the rinse bottle and deionized, distilled water, thoroughly rinse the storage container and the graduated flask. Place the plastic cover back on the graduated flask.

(e) Complete the history form. Fill out a sample identification label, as shown in Figure 11, and stick it onto the sample bottle.

(f) Put both the labelled sample bottle and the completed history form in a mailing container. Secure top and bottom of mailing container with strong tape and send to analytical laboratory for chemical analysis.

CANSOC

STATION SOMEPLACE

MONTH JULY YEAR 1980
MOIS ANNÉE

COLLECTOR SANGAMO TYPE A
COLLECTEUR

Figure 11. Example of a label for a precipitation sample.

9.3.4 *Prevention of Sample Contamination*

Careful handling of equipment and samples to prevent contamination is extremely important. The dissolved substances have a very low concentration and any contamination will produce erroneous results.

Do not touch those surfaces which will come in contact with the precipitation samples, i.e., inside surface of the collection vessel.

9.4 Precipitation Sampling for Organic Analysis

9.4.1 *Sample Handling Procedures*

Samples to be analyzed for organic constituents are collected in square stainless steel buckets. At the end of the month, the precipitation sample must be removed from the collector and shipped to the laboratory. The following procedures must be employed:

(a) Remove the bucket from the collector and carry to a clean area free from dust, gasoline, oil, and any other materials that might contaminate the sample.

Note: Hands must be clean. Do not touch those surfaces which will come in contact with the precipitation samples (i.e., inside surface of the collection bucket). If the sample is frozen, i.e., snow or ice, let it melt at room temperature before proceeding.

(b) Using the corner of the square bucket as a pour spout, transfer the sample from the bucket to the pre-cleaned bottle, or bottles if required. Cap the bottle(s) tightly.

(c) Return the bucket immediately to the sampler. Do not rinse out the stainless steel sampler.

(d) Complete the Sample History Form, making one carbon copy. Fill out a sample identification label, an example of which is shown in Figure 11, and stick it onto the sample bottle(s).

Using a mailing carton and pre-addressed mailing sticker, send the bottle and the Sample History Form to the laboratory for analysis.

9.4.2 *Precautions*

It is essential that all operations be carried out in as clean a manner as possible. The following contaminants should be strictly avoided:

(a) gasoline
(b) oil
(c) cigarette smoke and ash
(d) organic liquids
(e) other volatile materials that can lead to contamination of the sample by vapours or smoke.

Materials such as dust and dirt should also be kept from samples.

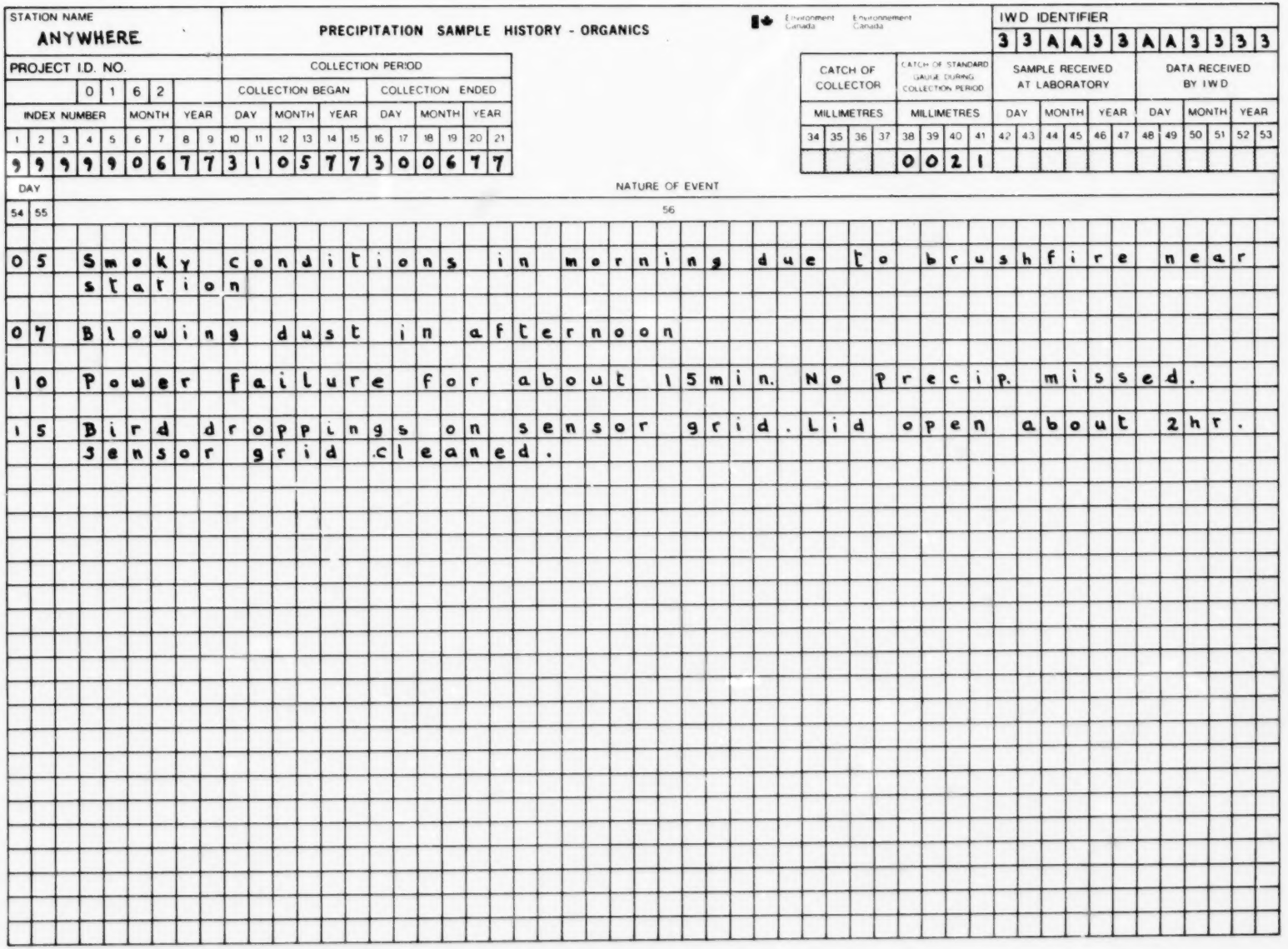
If it is suspected that the sample has been contaminated with any of these materials, then it should be reported on the Sample History Form.

9.5 Records of Precipitation Samples

The interpretation of the results of the chemical analysis will depend to a large extent on any phenomena affecting the chemical constituents or the physical collection of precipitation. Thus, it is necessary to maintain a carefully prepared record pertaining to each sample. A Precipitation Sample History Form is provided for this purpose.

9.5.1 *Completing the Precipitation Sample History Form*

Both the Water Quality Branch and the Atmospheric Environment Service use a similar Sample History Form, which is described below. An example of a completed form is shown in Figure 12.



STATION NAME: ANYWHERE

PRECIPITATION SAMPLE HISTORY - ORGANICS

Environment Canada / Environnement Canada

IWD IDENTIFIER: 3 3 A A 3 3 A A 3 3 3

PROJECT I.D. NO.							COLLECTION PERIOD											
			0	1	6	2		COLLECTION BEGAN			COLLECTION ENDED							
INDEX NUMBER					MONTH		YEAR	DAY		MONTH		YEAR	DAY		MONTH		YEAR	
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21
9	9	9	9	9	0	6	7	7	3	1	0	5	7	7	3	0	0	6	7	7

CATCH OF COLLECTOR				CATCH OF STANDARD GAUGE DURING COLLECTION PERIOD				SAMPLE RECEIVED AT LABORATORY						DATA RECEIVED BY IWD					
MILLIMETRES				MILLIMETRES				DAY		MONTH		YEAR		DAY		MONTH		YEAR	
34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53
				0	0	2	1												

DAY		NATURE OF EVENT
54	55	56
05		Smoky conditions in morning due to brushfire near station
07		Blowing dust in afternoon
10		Power failure for about 15 min. No precip. missed.
15		Bird droppings on sensor grid. Lid open about 2 hr. Sensor grid cleaned.

Figure 12. Example of a completed precipitation sample history form.

The information listed below must be entered in the space provided. Make one carbon copy in addition to the original.

Heading	Columns	Coding
Station name	-	Enter station name
Type of collector	-	Enter full name (e.g. Sangamo Type A)
Index number	1-5	Insert the 5-digit index number of the station, e.g., block and station number
Month, year	6-9	Enter the month and year over which the sample was collected, e.g., June 1975 would be 0 6 7 5
Day, month, year	10-15	Enter the day, month and year that collection of the sample began, e.g., 01 June 1975 would be 0 1 0 6 7 5
Day, month, year	16-21	Enter the day, month and year that collection of the sample ended, e.g., 30 June 1975 would be 3 0 0 6 7 5
Sample volume	30-33	Enter volume of sample to the nearest 10 mL. If volume is less than 200 mL leave these spaces blank and make an entry in the "Nature of Event" space to this effect
Catch of collector	34-37	Leave blank
Catch of standard	38-41	Enter four digits for the catch of the standard precipitation gauge during the collection period defined by column 10-21. Catch is recorded as water equivalent to the nearest *whole millimetre*, e.g., 76 mm would be 0 0 7 6
Sample received by laboratory	42-47	Leave blank
Date received by laboratory	48-53	Leave blank
Day	54-55	Enter two digits for the day that any unusual event occurred which could affect the sample
Nature of event	56	Enter a brief statement on the nature of the event which occurred

Events of particular interest follow:

(a) High winds and/or unusual amounts of blowing dust.

(b) Blowing snow, i.e., snow particles raised by the wind to sufficient heights above the ground to reduce the horizontal visibility at eye level to 10 km or less.

(c) Missed precipitation events with an estimation of amount missed, whenever possible.

(d) Snow capping of collection vessel.

(e) Objects found in collection vessel. Indicate nature of object, such as leaf, insect, paper, bird droppings, etc.

(f) Power failure.

Note: If objects are found in the collection vessel, do not attempt to remove them, as further contamination may result. If any other event occurs which may affect the precipitation sample, it should be entered on the Sample History Form.

Sediment Sampling Procedures

Sediment plays an important role in water quality. Part of the assimilative capacity of natural water systems for metals, pesticides and herbicides is the ability of the sediment to bind these substances, thus removing them from the water. On the other hand, many toxic substances stored in the sediment are released to the surrounding waters by a variety of chemical and biochemical reactions, thus making them available to the organisms living in these waters. Lake and stream sediments often reflect recent additions of heavy metals before elevations of such elements are detectable in the overlying water. While water analysis may therefore indicate no elevated concentrations in the soluble phase, a water body may still be heavily polluted with organic and inorganic material in sediments. The possibility of reentry of sedimented material into the water, owing to physical, chemical, or biological processes in natural situations, always exists. Also, with bottom-feeding organisms, sediments may be more important than water as a source of organic and inorganic substances.

To collect valid suspended sediment samples, samplers and sampling procedures must be designed to represent accurately the water/sediment system being studied. The procedures and apparatus employed for sediment sampling depend on the type of sediment being sampled. The methodology and the equipment used for sampling suspended sediments are different from those required for sediment deposits.

10.1 Sample Collection

Suspended sediment samples are collected to determine the quantity as well as the physical and chemical characteristics of those sediments in suspension. On the other hand, bottom sediments are sampled to provide the physical and chemical characteristics of those particles that make up the bed of the system being studied at specific locations.

10.1.1 *Sample Integrity*

For some purposes bed sediment samples can be disturbed, i.e., the individual particles can be rearranged relative to each other and it is unimportant that the volume and shape of the sample be altered from the actual conditions of the deposit. However, for most purposes undisturbed samples are required. When the purpose of sampling is to obtain information related to the vertical composition of the deposits or on the distribution of contaminants from a certain depth, undisturbed core samples must be taken.

10.1.2 *Samplers*

10.1.2.1 Suspended Sediment Samplers

Samplers used for suspended sediments must allow the collection of a sample representative of the water-sediment mixture at the sampling point or sampling zone at the time of sampling. Samplers are of three general types:

(1) integrating samplers,
(2) instantaneous or grab samplers, and
(3) pumping samplers.

Standard suspended sediment samplers used to sample streams and rivers should not be used in lakes, reservoirs or other bodies of water where water is stationary or almost stationary. In such conditions, samplers which operate on principles similar to the grab type samplers are used (Section 3.3.2).

10.1.2.2 Bottom Sediment Samplers

10.1.2.2.a Core samplers

Gravity and piston corers are used to collect undisturbed samples of river, lake, reservoir and pond deposits. Samplers of this type are essentially tubes which are forced into the bed of the system. A few such samplers are illustrated in Figure 13. Samples are retained inside the barrel of the sampler on retrieval by a partial vacuum formed above the sample and/or by a core retainer at the lower end.

10.1.2.2.b Grab samplers

Grab samplers are more commonly used than core samplers for collecting deposited sediments, as they are often much lighter and in some circumstances much easier to use. If properly used, the grab sampler encloses a volume of the bed material and isolates the sample from water currents during its ascent to the surface to yield a reasonably good undisturbed sample.

The *Shipek grab sampler* consists of a steel shank, weight and a spring loaded bucket. The sampler (Fig. 14) is designed to collect relatively undisturbed samples of bottom surface sediments. The steel bucket, a half cylinder with closed ends, is lowered through the water column in an inverted position (convex upward) until it contacts the sediment surface. The internal weight mounted above it continues to drop and triggers a release mechanism which frees the inverted bucket. The bucket is then forced to rotate 180° on its axis at high speed by a pair of externally mounted helical springs. The bucket then cuts in a continuous curve through the sediment until it is stopped in a position concave upwards. In most cases the rotational shear is far greater than the sediment shear strength, and consequently the cutting action is very clean (producing minimal disturbance) particularly in soft clays, muds, silts and sands. The bucket is released by pulling outwards on its pivot pins and a clean-cut undisturbed bottom sample is recovered.

The Shipek sampler functions well as a result of the mechanism which permits the bucket to close with its separation plane aligned in the horizontal rather than in the vertical. Good samples can still be retrieved even when bucket closure is prevented by pebbles or similar material, 2 to 5 cm across. With the bucket properly rotated, sample washout is completely avoided and the body of the sampler protects the bucket from washout on ascent.

The *Mini-Shipek sampler* works exactly as the standard Shipek described above except, as the name implies, takes a considerably smaller sample. This sampler is used basically from small boats by hand.

The *Birge-Ekman dredge* (Fig. 15) is available in a number of models, all basically similar in design. The standard model consists of a brass or stainless steel box with a pair of free-moving hinged flaps. During descent,

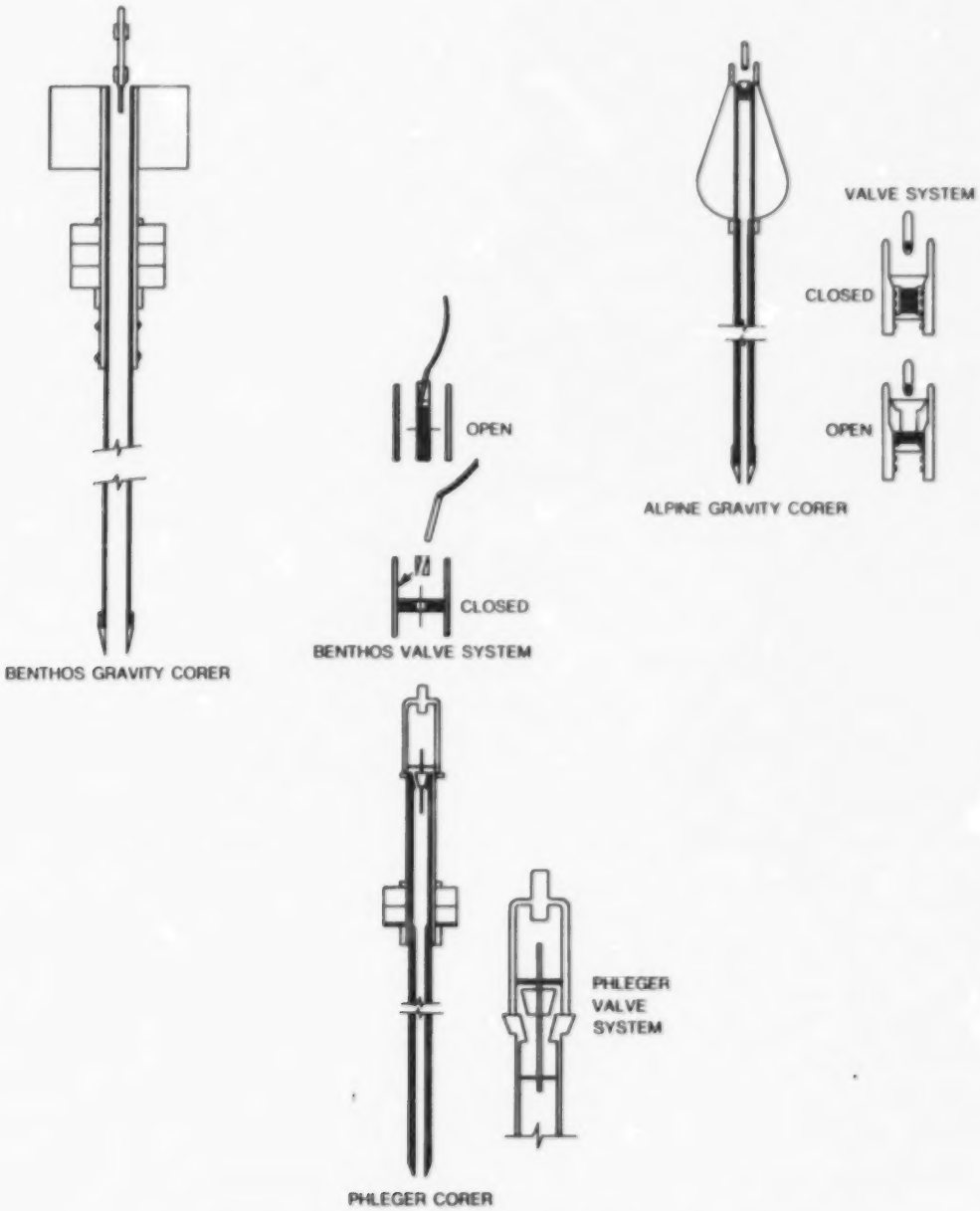


Figure 13. Examples of sediment corers (from Mawhinney and Bisutti, 1981).

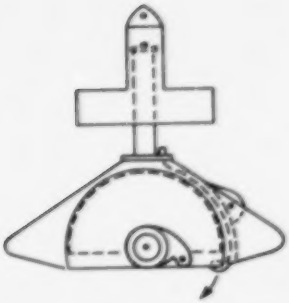


Figure 14. Shipek grab sampler (from Mawhinney and Bisutti, 1981).

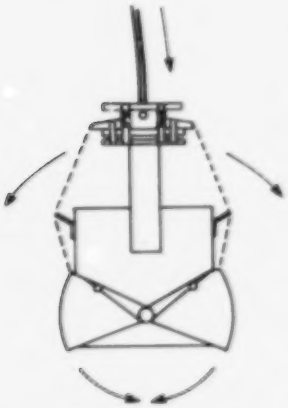


Figure 15. Birge-Ekman dredge (from Mawhinney and Bisutti, 1981).

these flaps are forced open by the pressure of water passing through the open box. On ascent, the flaps cover the upper surface of the box and prevent disturbance of the material inside it. Pivot points on opposite sides of the box serve as mounting points for a pair of spring-tensioned scoop-like jaws. The jaws are held open by wires which lead up to an externally mounted trigger assembly. This is normally triggered by a surface release messenger weight. The jaws are designed to overlap to prevent washout during retrieval. The dredge is in the form of a box with sides 15.2 cm long. With the jaws closed, the maximum depth of their cut is about 4.5 cm. The standard Birge-Ekman weighs 4.5 kg or less, and the maximum sample surface area is approximately 230 cm^2 with a maximum sample capacity of about 3900 cm^3.

The Birge-Ekman dredge is suitable for soft clays, muds, silts and silty sands. The sampler should be used during calm water conditions, typically in small lakes or restricted areas. The lack of sample disturbance, square cross section and moderate penetration make this sampler suitable for detailed studies, e.g. biological and geochemical, of the top 2-3 cm of bottom sediment. It is lightweight and easy handling makes it well suited to small boat operations.

The sampler performs well except when used in very coarse or shelly sediment. Under these conditions, material may be trapped between the jaws, preventing their closure. In this case washout may be severe. Due to the overlapping of the jaws, a slight imperfection of closure can be tolerated.

10.1.3 *Site Location*

10.1.3.1 Rivers

In instances in which sediment transport data are required, it is necessary to locate the sampling sites near a water quantity gauging station so that accurate stream discharge information is available at all times. Sampling locations immediately upstream from confluences should be avoided, as they may be subjected to back water phenomena. In streams too deep to wade, it may be advantageous to locate sampling sites under bridges or cableways. When sampling from bridges, the upstream side is normally preferred. Sampling on the downstream side of the bridge presents limited upstream visibility, and care must be taken to avoid sampling in areas of high turbulence, near the piers. Sediment samples collected near piers are often unrepresentative of the general sediment transport characteristics. Also attention must be paid to the accumulation of debris or trash on the piers, as this can seriously distort the flow and hence the sediment distribution.

Sampling sites should be accessible during floods, since sediment transport rates are high during these times.

It is important that the same transect be used during the entire sampling period so that the variability associated with the sampling procedure is minimized.

10.1.4 *Sample Handling and Preservation*

Procedures for handling and preserving sediment samples depend on the specific analyses needed and on whether the sample is from the suspended or bottom environment. Samples for trace metal analyses require special precautions to prevent contamination and also require preservation. Collection bottles should be precleaned by thoroughly washing, as outlined in Section 3.5.2.

10.1.4.1 Suspended Sediment

As soon as possible after collection the sample should be filtered. The filtrate can then be used for measuring the dissolved constituents. Preservation procedures, as outlined in Section 6.2, are followed.

Suspended sediment sample analyses are often limited on account of the difficulty in obtaining sufficient sediment for the many subsamples required for the different analyses. A composite of a large number of representative samples may be necessary.

10.1.4.2 Bottom Sediment

Samples of sediment for routine particle size analysis can be transported and stored without refrigeration. Table 7 lists a number of parameters and methods of preservation for bottom sediment samples.

Shipping of Samples

Once collected, water samples must be transported to the laboratory. The mode of transport will depend upon the geographic location and on the maximum permissible time lapse before analysis for each parameter. The field investigator is responsible for delivering the samples to the air, bus or train terminal on schedule so that there will be minimal delay in sample transport. Logistics for sample shipping and storage should be determined before fieldwork is initiated.

11.1 Preparation for Shipping

During packing, do not separate the samples from one location into different shipping boxes or coolers unless all bottles of one size must be shipped together because of container size.

If samples from one station must be divided and placed in two cartons, a copy of the field sampling sheet pertaining to the bottles must be enclosed in each box. (See Section 5.2 for description of field sampling sheets.) This could require two or more field sheets (carbon paper or similar system is useful). In addition, a clear identifier label must be attached to each sample (see Sections 11.2 and 11.3 for details on labels).

Before shipping, always check that all sample bottles recorded on the field sampling sheets have been placed in the carton. Indicate the shipping date and mode of transport on the field sampling sheet.

Preparation for shipping should follow these steps:

(a) Fold the field sampling sheets and place them in plastic bags. The bags would protect the field sheets should spillage occur during transport.

(b) Pack samples well to prevent breakage, affix identification, destination and FRAGILE labels to each carton or cooler. (Section 11.2 contains a description of shipping labels.) Mark top of cartons or coolers as THIS END UP in red ink. Also, number the boxes or coolers in a shipment, e.g., 1 of 4, 2 of 4, 3 of 4, and 4 of 4. This informs the receiver of the number of pieces to expect.

(c) Keep a list of samples packed in each carton or cooler.

The investigator should also retain a copy of the field sampling sheets.

11.2 Shipping Labels

Shipping labels which clearly identify the source and destination of the sample bottles increase the efficiency of sample shipping. An example of a shipping label is provided here.

FROM: John Doe
351 St. Joseph Blvd.
Hull, P.Q. K1A 0E7

SHIP TO: P.O. Box 5050
867 Lakeshore Dr.
Burlington, Ontario
L7R 4A6

DATE:
FEB. 12/81

ATTN: Jim Smith

All shipping labels should include a clearly written, precise destination address, including postal code. There should be no delay in shipping on account of incomplete addressing. If a particular person is in charge of the analytical side of the project, the label should be marked to his or her attention. Also included on the shipping label is the sender's name and address, and the date of shipping.

Shipping labels must be clearly legible on all boxes. Preprinted or pretyped self-sticking labels are recommended.

11.3 Labels on Individual Bottles

Each bottle should be clearly marked. As a minimum, each label should state:

(a) the station code number; a narrative description is optional,

(b) sampling date and time,

(c) parameters that are to be analyzed from that specific bottle; prearranged codes can be used to indicate certain groups of parameters, and

(d) whether or not the sample is preserved and with what. Coloured labels can be used to indicate the preservation method.

A number of methods may be used to label individual bottles. Labels may be applied directly to each bottle with a water-insoluble marker or they may be written on masking tape, which is then attached to the sample bottle. Information may also be written on commercially available tags and then attached to the bottle with elastic bands, string or tape. However, it is recommended that elastic bands not be used on sample bottles for metals or metal blanks.

Field Safety

Samples are collected during a wide range of weather extremes. Knowledge of the hazards that may be encountered and the means by which they can be minimized is a necessary consideration in any field project. Field personnel must ensure that they are adequately equipped before they embark on a sampling trip. As a precautionary measure, survival kits and first aid kits are recommended, especially for personnel operating in isolated areas or in regions subject to rapid weather changes. The collection of surface water samples may involve collection from bridges, docks, boats, or by wading and may include drilling through ice. The inability to swim is a disadvantage.

Some field safety procedures are given in the following sections. In addition, the Water Quality Branch proposed field safety procedures are given in Section 12.7.

12.1 Safety Precautions when Sampling from Bridges

Traffic may present serious problems when working from bridges. Bridges with sidewalks for pedestrian traffic provide a margin of safety. If it is necessary to interfere with traffic, suitable arrangements must be made in advance with local authorities. In addition, warning lights and "men working" signs should be erected.

The field technician must take special care when sampling from bridges over navigable water, as boat operators and water skiers may not be able to see apparatus suspension lines. Samples should be taken in the absence of water traffic, or special precautions such as the attachment of flags etc. should be taken to make suspension lines visible.

Power lines strung close to bridges are also dangerous. If samples are going to be repeatedly collected from a bridge near a power line, a warning sign indicating the presence of the power line should be installed. A hazard warning should also be noted on the site location form.

12.2 Safety Precautions when Sampling from Boats

Water samples are frequently collected from boats. As with cars and trucks, service checkups are essential. Gasoline, spare parts (particularly shear pins, spark plugs) and a tool box must be checked and included on each field trip. Field personnel, when sampling in remote areas, should take along emergency rations. A spare motor is also recommended when sampling in remote areas or over large bodies of water or fast-flowing rivers. Life jackets are necessary equipment; these and other buoyancy devices must conform to the approved safety requirements outlined by Transport Canada in the Canadian Coast Guard Boating Handbook. When sampling during winter months, extra clothing, parkas, boots, gloves, etc., should be available.

All aspects of boat operation must conform with the regulations provided by Transport Canada in the Boating Handbook. In addition, local or regional police boating regulations must be followed at all times. It is always advisable to check the weather forecast before planning an extended sampling trip. Sampling should be curtailed when weather conditions make it unsafe to continue.

To ensure smooth operation and to conserve time in the field, it is important to establish boat launching sites at marinas, public piers, etc., in advance of the actual sampling program.

Boat operations on flooded streams are particularly hazardous. During these periods the boat operator must cope with the fast current and also be on the alert for shallows and for floating and submerged debris. Floating ice is very dangerous; even when there is no danger to the boat, changes in wind, current or tide may shift the position of ice flows and cut the boat off from shore.

12.3 Safety Precautions while Wading

Wading is one of the easiest methods to collect samples from many streams, and it may also be extremely dangerous. Wading permits the investigator to examine stream flow and decide where to sample. Rubber boots or even chest-high waders are standard equipment. A wading rod or similar probing instrument is often useful to estimate the current and to locate holes and unsafe footing. If the wader has any uncertainty about his ability to wade a stream, he or she should be attached by a rope to a rigid mooring and must wear an approved flotation device. An extra change of clothing is always advisable, and essential during the colder months.

12.4 Safety Precautions when Sampling through Ice

Considerable risk may be involved in collecting samples through ice. A hole of sufficient diameter to collect a sample from a bridge may be broken in thin ice by dropping a weight attached to a handline. Generally, it is not advisable to collect samples near or off a bridge because the samples may become contaminated with road de-icing agents.

The ice in a stream is likely to be of variable thickness, and the strength of the ice cannot be estimated from the apparent thickness near the edges. In advancing across an ice-covered stream, it is advisable to test the ice with an ice chisel every few steps. It may also be hazardous to collect samples downstream from bridge supports, as the ice may be thin as a result of modified flow patterns and de-icing agents from the road surface. A few inches of new snow can conceal thin ice. Areas with rapids should be approached with extra caution because ice is usually much thinner due to the water movement. Working

from ice during breakups is particularly hazardous. Vehicles should never be driven over ice-covered bodies of water except where winter ice roads exist, and then with caution.

Winter sampling requires suitable clothing to maintain a comfortable working temperature and to prevent frostbite. A warm hat, woollen undergarments, extra socks, mitts, proper footwear, and in some cases glasses to prevent snow blindness, should be taken. Before leaving the base, the field investigator should also check the wind chill factor because reports of temperature alone may be deceiving.

12.5 Safety Precautions when Handling Chemicals

Acids and bases used for the preservation of water samples should be stored and handled with care. Care must be exercised to avoid inhalation of vapours or direct contact with skin, eyes and clothing. Acids and bases should never be pipetted orally. They must be securely stored during transport to prevent spillage. Spills should be cleaned up immediately by dilution with large quantities of water, neutralization, or mopping up of the chemical followed by disposal of the contaminated material. It is recommended that gloves and an apron be carried in the field.

Skin which has been in contact with acids or bases should be washed immediately with plenty of water. After the skin has been washed, the contaminated area may be swabbed with a neutralizing solution. This procedure should be followed by a second washing with soap.

If any chemicals enter the eyes, they should be rinsed *immediately with plenty of water.* Rinse the outside of the eyes as well. It may be necessary to hold the eyelids open during the wash procedure. Continue rinsing for several minutes. After first aid, all eye injuries must be treated professionally.

Routine care should be taken to prevent bottle and other glassware breakage during sampling; for example, overtightening of tops, rough handling of pipettes, and improper packaging and storage of glass sampling bottles may result in breakage during transport.

12.6 Survival Kit and Rations

The components of a typical survival kit are listed below. It should be noted that other articles may be beneficial and should be included as required.

Survival kit	Survival rations
Thermal blanket 1.8 m x 2.3 m	Compressed food packets (1200 calories each)
Sleeping bag	Dry soup mix
Tent	Coffee
First aid kit	Tea
Single-burner folding stove	Powdered milk
Solid cooking fuel	Sugar
Flashlight (disposable)	

Survival kit (continued)
Flares
Small signal mirror
Candles, 15 h with holder
Windproof matches
Wool socks
Wool gloves
Balaclava
Knife, fork, spoon
Metal mug
Tissues
Plastic garbage bags (orange)
Gas line antifreeze

12.7 Proposed Water Quality Branch Field Safety Procedures

This section contains a series of field safety procedures for the use by WQB personnel. Although at the time of writing of this manual, March 1982, this set of procedures represents the consensus of the Branch, the procedures are still at the *proposal* stage. Before being adopted by the WQB they must be submitted to the Department of Labour and then to the Treasury Board for approval. *Until this approval, the Treasury Board standard TB STD 3-14 entitled "Personnel Protection Equipment Safety Standard," Section "Drowning Hazards" (in "Handbook of Occupational Health and Safety," published by the Treasury Board, Ottawa, 1978) must be followed.* This standard states the following:

> "42. No employee shall work or be permitted to work over water or at any other work location where there is a risk of drowning unless
>
> (1) the employee is wearing an approved life jacket or other buoyancy device of a type described in paragraph 43; or
>
> (2) the employee is prevented from falling into the water by a safety net or platform or a safety restraining device; and
>
> (3) the employee is accompanied by at least one other person."

This standard applies to all Public Service departments and agencies. The Department of Labour is responsible for enforcing the standard.

12.7.1 Proposed WQB Field Safety Procedures

A. Operations Requiring Boats

(1) Whenever work is carried out from a boat, at least two persons must always be present, wearing approved flotation devices and operating in accordance to water safety rules and regulations.

(2) In situations where only one WQB person is available for fieldwork which requires two or more persons, the assistance of a person who is not from WQB must be obtained. However, care must be exercised when choosing field assistants so that they do not cause safety problems.

(3) Only boats and equipment suitable for the water system being sampled are to be used. The outboard motor used and its operation must comply with Transport Canada Coast Guard Regulations.

(4) Care must be taken to prevent overloading boats with sampling equipment. Equipment requirements for each boat will depend on its size and type and should comply with "Transport Canada's Small Vessel Regulations."

(5) In isolated locations, on large water bodies and where there are downstream dangers such as rapids or waterfalls, a spare motor should be carried.

(6) Before setting out onto the water, the operating condition of the boat must be checked and any irregularities corrected.

B. Procedures when Working on Ice Covered Lakes and Rivers

(1) Whenever work is carried out on ice, two persons must work together employing safety lines and other equipment, e.g. two ice spikes, related to safe working practices on ice. Winter safety flotation devices must be worn.

(2) Field personnel must follow departmental guidelines respecting safe operations on ice, e.g. "Information and Guidelines Respecting Safe Operations over Ice," Public Service Commission, December 1976.

(3) Field personnel must learn how to recognize ice conditions and to proceed accordingly. However, care must be exercised, as ice surface often does not relay information related to its thickness.

(4) Field staff are to be cautious when approaching ice areas covered with snow.

C. General Practices

(1) In situations where there is only one man in the field performing duties not associated with sampling from boats or on the ice, a safety line must be attached to a rigid mooring and an approved flotation device worn.

(2) Water depth during wading operations should be checked with a pole before steps are taken.

(3) When wading equipment is worn, the support straps must be outside the clothing.

(4) In all situations field parties are required to leave accurate sampling schedules and expected itineraries in the office.

(5) Field staff must be provided with available information concerning characteristics of water bodies under study.

(6) Sampling should never be carried out in weather which is considered by the party leader or party member to be hazardous to the well-being of the field staff and/or equipment.

(7) Field staff must take a water safety course, including water survival and field first aid instruction. Whenever possible, field personnel must attend courses related to other safe field practices, e.g. wilderness survival and basic mechanics. Refresher courses approximately every three years are also recommended.

Glossary

Absorption - the incorporation of a substance into the body of another.

Accuracy - refers to the agreement between the measured value and the accepted or "true" value. It is expressed as the difference between these two values.

Acid extractable metal - the concentration of a metal in solution after treatment of an unfiltered sample with dilute mineral acid, cold or hot.

Acidity - the quantitative capacity of aqueous media to react with hydroxyl ions.

Acid mine drainage - low pH drainage water from certain mines. The low pH is usually caused by the oxidation of sulphides to sulphuric acid. Mine drainage can also contain high concentration of metal ions.

Adsorption - the surface retention of solid, liquid or gas molecules, atoms or ions by a solid or liquid.

Algal mat - a moribund surface algal population.

Algicide - a chemical used to kill algae. Aligicides are often applied to water to control nuisance algal blooms.

Aliquot - a representative sample of a larger quantity.

Alkalinity - a measure of a water's capacity to neutralize an acid. It indicates the presence of carbonates, bicarbonates and hydroxides, and, less significantly, borates, silicates, phosphates and organic substances. It is expressed in milligrams of $CaCO_3$ per litre.

Ambient temperature - the temperature of the surrounding medium, such as ambient air, which comes into contact with the apparatus.

Analytical grade - a chemical with a level of purity high enough to permit its use in precise analytical determinations.

Aquatic life - those organisms found living or growing in, on or near water.

Bedload - material moving on or near the stream bed by rolling, sliding, and sometimes making brief excursions into the flow a few diameters above the bed.

Bedload discharge - the quantity of bedload passing a transect in a unit of time.

Bedload sampler - a device for sampling the bedload.

Bed material - the sediment mixture of which the bed of the water body is composed. In alluvial streams bed-material particles are likely to be moved at any moment.

Bed-material sampler - a device for sampling bed material.

Benthos - bottom-dwelling organisms.

Biochemical oxygen demand (BOD) - a measure of the quantity of oxygen used in the biochemical oxidation of organic matter in a specified time, at a specified temperature, and under specified conditions. The standard measurement is made for 5 days at 20°C. In general, a high BOD indicates the presence of a large amount of organic material. It is normal to find a BOD of 2 to 3 mg/L in river waters receiving natural drainage.

Biodegradability - the characteristic of a substance that can be broken down by microorganisms.

Biodegradation - the process of destruction or mineralization of either natural or synthetic materials by the microorganisms of soils, waters or wastewater treatment systems.

Biomass - the weight, wet or dry, of all living matter in a specified area or volume of environment, for example, a square metre of stream bottom.

Birge-Ekman dredge - a sampler designed for the collection of the top 2-3 cm of bottom sediment. This apparatus is suitable for soft clays, muds, silts and silty sands.

Blank - a sample of distilled water.

Bloom - a clearly visible concentrated growth or aggregation of plankton (plant and animal) in a water body.

Bottom sediment - those sediments which make up the bed of a body of running or still water.

Centroid of flow - midpoint of a channel of uniform flow.

Chemocline - a zone of rapid change, with depth, in the concentration of dissolved substances in a lake. The boundary between the circulating and non-circulating layers of a lake.

Coliform bacteria - a group of bacteria predominantly inhabiting the intestines of man or animals, but also occasionally found elsewhere. Fecal coliform bacteria are those organisms associated with the intestine of warm-blooded animals, and they are used commonly as an indicator of the presence of fecal material and the presence of organisms potentially capable of causing disease in man.

Colony - a group of individual animals or plants living together and dependent on one another to a varying degree. In some colonies the individuals are structurally connected and function as a single unit, as in many algae.

In other colonies the individuals are not structurally connected but often, notably in insect colonies, show a high degree of social organization. Also, a growth of a particular type of bacterium on a culture medium.

Community - in ecology, a collection of populations of animals and plants that occur naturally together in a common environment, such as the organisms inhabiting a woodland. The individuals and populations within the community interact with one another and with the abiotic (non-living) surroundings, with the community and abiotic surroundings together constituting an ecosystem. Communities vary considerably in size and one large community may contain a number of smaller ones.

Composite sample - a sample obtained by mixing several discrete samples, or representative portions thereof, into one bottle (see flow proportional and sequential composite samples).

Contamination - a foreign or unwanted material which renders a sample unfit for meaningful analyses.

Cosmic-ray-produced nuclides - short-lived radioisotopes such as tritium, beryllium-7 and carbon-14 formed by the continuous "rain" of electrons and nuclei of atoms from space interacting with certain atmospheric and terrestrial elements.

Cross section - a plane perpendicular to the axis of flow.

Deionized water - water that has passed through a column to remove charged, usually inorganic particles.

Depth-integrated sample - a sample which represents the water-suspended sediment mixture throughout the water column so that the contribution to the sample from each point is proportional to the stream velocity at that point.

Detection limit - the smallest concentration of a substance which can be reported as present with a specified degree of precision and accuracy by a specific analytical method.

Deterioration - a decline in the quality of a sample over a period of time due to improper preservation techniques.

Detritus - finely divided non-living settleable material suspended in the water: organic detritus, from the decomposition of the broken down remains of organisms; inorganic detritus is settleable mineral materials.

Discharge - the flow rate of a fluid at a given instant expressed as volume per unit of time (see stream discharge).

Dissolved concentration - the concentration of a constituent of an unacidified water sample that will pass through a 0.45-μm membrane filter. The filtration should be done immediately after sample collection.

Dissolved load - the part of the stream load that is carried in solution, such as chemical ions yielded by weathering and erosion of the land mass.

Dissolved oxygen (DO) - is the amount of oxygen dissolved in a given volume of water.

Drainage basin - the area tributary to or draining to a lake or stream (see also watershed).

Duplicate samples - obtained by dividing one sample into two or more identical sub-samples.

Ecology - the study of the relationships of organisms to their environment.

Ecosystem - a functional system formed by the organisms of a natural community and their environment.

Epilimnion - the uppermost layer of water in a lake, characterized by an essentially uniform temperature that is generally warmer than elsewhere in the lake and by a relatively uniform mixing caused by wind and wave action. Also the light (less dense) oxygen-rich layer of water than overlies the metalimnion in a thermally stratified lake.

Eutrophication - the process of over-fertilization of a body of water by nutrients that produce more organic matter than the self-purification processes can overcome.

Extractable metal - see acid extractable metal.

Filterable constituent - see dissolved constituent.

Filtrate - the fluid that has passed through a filter.

Filtration - the process of passing a liquid through a filter to remove suspended matter.

Floc - small masses formed in a fluid through coagulation, agglomeration or biochemical reaction of fine suspended particles.

Flocculant or flocculating agent - a substance which, when added to water, forms a floc, thus expediting the settling of suspended matter; for example, alum, ferrous sulphate or lime.

Flow proportional composite sample - is obtained by (1) continuous pumping at a rate proportional to the flow; (2) mixing equal volumes of water collected at time intervals which are inversely proportional to the volume of flow; (3) mixing volumes of water proportional to the flow collected during or at regular time intervals. This sample will indicate a "flow" average water quality condition over the period of time of compositing.

Fluvial characteristics - of or pertaining to a river or rivers; existing, growing or living in or about a stream or river; produced by the action of a stream or river.

Gauging station - a selected location on a stream channel where discharge and other related parameters are measured continuously or periodically.

Geochemistry - a science that deals with the chemical composition of and chemical changes in the crust of the earth.

Geohydrology - the science involving underground water, often referred to as hydrogeology.

Glass distilled water - water that has been freed from dissolved or suspended matter by distillation in a glass system.

Grab sample - a sample taken at a selected location, depth and time.

Groundwater - all subsurface water.

Hardness, total - the sum of calcium and magnesium concentrations, both expressed as $CaCO_3$, in milligrams per litre. Other ions such as iron, manganese and aluminum contribute to total hardness, although they are usually present in much smaller concentrations and thus do not normally make a significant contribution to the total hardness. The hardness relates to a water's capability to precipitate soap or to produce lather from soap. The harder the water, the more difficult it is to lather soap. Hardness is normally expressed in milligrams $CaCO_3$ per litre. Waters of various hardness are classified as follows:

Hardness of 1 to 60 mg/L as $CaCO_3$ - soft water.
Hardness of 61 to 120 mg/L as $CaCO_3$ - medium-hard water.
Hardness of 121 to 180 mg/L as $CaCO_3$ - hard water.
Hardness >180 mg/L as $CaCO_3$ - very hard water.

Heavy metals - metallic elements with specific gravities greater than 5, such as cadmium, copper, lead and zinc.

Herbicide - substances or a mixture of substances used to destroy specific vegetation.

Homogeneous - uniform in composition

Hypolimnion - the lowermost layer of water in a lake, characterized by an essentially uniform temperature (except during a turnover) that is generally colder than elsewhere in the lake, and often by relatively stagnant or oxygen-poor water. Also the dense layer of water below the metalimnion in a thermally stratified lake.

In situ measurements - measurements made directly in the water body.

Kemmerer sampler - a messenger-operated vertical point sampler for water-suspended sediment.

Macro organisms - plant, animal or fungal organisms visible to the eye.

Macrophytes - large plants.

Metalimnion - see thermocline.

Membrane filter - a specially prepared filter of cellulose ester with a controlled pore diameter.

Microphyte - a microscopic plant; a dwarfed plant due to unfavourable environmental conditions.

Molarity - the concentration of a solution expressed as the number of gram-molecular weights of a substance dissolved in 1 L of solution.

Multiple sampler - an instrument permitting the collection of several water-suspended sediment samples of equal or different volumes at each site, simultaneously.

Non-point waste source - a general, unconfined waste discharge.

Normality - the concentration of a solution expressed as the number of gram-equivalent weights of a substance dissolved in 1 L of solution. Normality is abbreviated by *N*.

Nutrient - a substance, element or compound necessary for the growth and development of plants and animals.

Oligotrophic waters - waters with a deficiency of nutrients and plentiful amounts of dissolved oxygen. They support little organic production.

Organic-free water - water free from organic substances.

Particle size - the dimension, such as average diameter or volume, of the particles in a sediment or rock, or of the grains of a particular mineral that make up a sediment or rock, based on the premise that the particles are spheres or that the measurements made can be expressed as diameters of equivalent spheres. It is commonly measured by sieving, by calculating settling velocities or by determining areas of microscopic images.

Periphyton - the association of aquatic organisms attached or clinging to stems and leaves of rooted plants or other surfaces projecting above the bottom.

Pesticide - a chemical agent that destroys pests.

pH - the negative $\log_{10}$ of the hydrogen ion activity in solution. Water with pH values between 0 and 7 is acidic, with pH value of 7 is neutral, and with pH values between 7 and 14 is alkaline.

Photosynthesis - the process by which simple sugars are manufactured from carbon dioxide and water in plant cells. It also requires the presence of chlorophyll and light.

Phytoplankton - plant microorganisms, such as certain algae, living unattached in the water.

Plankton - organisms of relatively small size, mostly microscopic, which either have relatively small powers of locomotion or drift in the water subject to the action of waves and currents.

Point sample - see grab sample.

Point wastes source - any discernible, confined and discrete conveyance such as any pipe, ditch, channel, tunnel or conduit from which pollutants are discharged.

Pollution - the condition caused by the presence of substances of such character and in such quantities that the quality of the environment is impaired (see water pollution).

Population - a generic term denoting any finite or infinite collection of individual things, objects or events in the broadest concept. In ecology, a group of individuals of the same type, particularly of the same species, within a community. Populations have certain characteristics not shown by individual organisms or the community as a whole. Characteristics of a population include density (size in relation to unit space), birth and death rates

(natality and mortality, respectively), age distribution, sex ratio, dispersion (distribution of the individuals within the area) and growth rate.

Precision - denotes the agreement between the numerical values of two or more measurements on the same homogeneous sample made under the same conditions. The term is used to describe the reproducibility of the measurement or method. It can be expressed by the standard deviation.

Preservative - a substance added to the sample in order to maintain given component(s) in a particular state, e.g. dissolved metals in solution.

Pumping sampler - a sampler with which the water-sediment mixture is withdrawn through a pipe or hose, the intake of which is placed at the desired sampling point.

Radioactive contaminant - a radioactive material which is present in places where it may harm life or property.

Radioactivity - the property possessed by some elements, e.g. uranium, of spontaneously emitting alpha, beta and gamma rays by the disintegration of their nuclei.

Radionuclide - an isotope of an element (nuclide) that exhibits radioactivity.

Range - the difference between the lowest and highest values in a set of data.

Replicate samples (spatial) - two or more samples taken simultaneously in a given cross section of the water body under study. They are used for measuring the cross-sectional variations in the water quality parameters.

Replicate sample (temporal) - two or more samples taken at the same place sequentially at specified intervals over a specific period of time. They are used to determine the uncertainty in various water quality parameters due to temporal variations.

Representative sample - a sample of a universe or population whose composition is expected to exhibit their average properties.

Reservoir - an impounded body of water or controlled lake where water is collected and stored.

Residue - material that remains after gases, liquids and some solids have been removed usually by heating up the sample at a specified temperature for a specified period of time.

Sampling iron - an iron frame designed to hold sampling bottles of different sizes.

Sampling vertical - a vertical line from the water surface to the bottom along which one or more samples are collected to determine various properties of the water body, such as the concentration of suspended sediment, nutrients and metals.

Secchi disk - a circular metal plate, 20-30 cm in diameter, painted in black and white quadrants. It is used to determine the transparency of a water body.

Sediment - solid fragmental material that originates from weathering of rocks and is transported or deposited by air, water or ice, or that accumulates by other processes, such as chemical precipitation from solution or secretion by organisms. The term is usually applied to material held in suspension in water or recently deposited from suspension and to all kinds of deposits, essentially of unconsolidated materials.

Sediment discharge - usually the mass or sometimes the volume of sediment passing a stream transect in a unit of time. The term may be qualified, for example, as suspended-sediment discharge, bedload discharge or total-sediment discharge.

Sediment load - the amount of sediment passing a cross section of a river, stream, etc., in a specified period of time (see also bedload and suspended sediment load).

Sediment sample - a quantity of water-sediment mixture or deposited sediment collected to characterize its properties.

Sediment transport - see sediment discharge.

Sediment yield - the total sediment outflow from a drainage basin in a specified period of time. It includes bedload as well as suspended load, and usually it is expressed in terms of mass, or volume per unit of time.

Sequential composite sample - a sample obtained either by continuous, constant pumping of water or by mixing equal volumes of water collected at regular time intervals. This sample will indicate an average water quality condition over the period of time of compositing.

Shipek sampler - an instrument designed to collect relatively undisturbed samples of bottom surface sediments (also Mini-Shipek).

Species - a unit used in the classification of plants and animals. Includes closely related, morphologically similar individuals that interbreed with each other to produce fertile offspring. Members of different species do not normally interbreed; if they do, the progeny are sterile. In binomial nomenclature each species has two names. The first is the name of the genus and the second is the specific epithet (trivial name). For example, in the species *Homo sapiens*, *Homo* is the generic name and *sapiens* is the specific epithet.

Species population - a group of similar organisms residing in a defined space at a certain time.

Specific conductance - a numerical expression of a water's ability to conduct an electrical current. It is measured in microsiemens per centimetre (μS/cm) at a standard temperature, usually 25°C. At a given temperature the conductivity of water depends on the concentration of ions in solution.

Split sample - a single sample separated into two or more parts such that each part is representative of the original sample.

Standard deviation - a measure of the dispersion or spread of data points around the mean value of the data set

obtained by repetitive testing of a homogeneous sample under specified conditions.

Standard crop - the biota present in an environment at a selected point in time.

Sterilization - the process of destroying all forms of microbial life on and in an object.

Stream discharge - the quantity of water passing a stream transect in a unit of time.

Supernate or supernatant - the liquid, e.g. water, above the surface of settled sediment.

Surface water - natural water bodies, such as rivers, streams, brooks and lakes as well as artificial water courses, such as irrigation industrial and navigational canals in direct contact with the atmosphere.

Suspended load - the part of the sediment load that is in suspension.

Suspended sediment - those constituents of an unacidified water sample that are retained by a 0.45-μm membrane filter. It can impart a cloudy appearance to the sample.

Suspended solids - see suspended sediments.

Teflon - a man-made plastic material inert to all chemical, reagents except molten alkali metals. It is used for laboratory and field equipment.

Thermocline - the layer in a thermally stratified lake, between the epilimnion and hypolimnion, in which the temperature decreases most rapidly with depth. The horizontal layer of water characterized by a rapid decrease of temperature and increase of density with depth; sometimes arbitrarily defined as the layer in which the rate of temperature decrease with depth is equal to at least 1°C per metre. Also known as metalimnion.

Time composite sample - see sequential composite sample.

Total concentration - the concentration of a given constituent determined in an unfiltered sample after vigorous digestion; the sum of the concentrations in both filterable and unfilterable (suspended) fractions.

Total dissolved solids (TDS) - the concentration of dissolved substances in a water, usually expressed in milligrams per litre.

Trace element - a chemical element found naturally or required by living organisms in extremely small quantities (micronutrient).

Transect - a cross section of a water body.

Turbidity - a measure of the optical property that causes light to be scattered and absorbed rather than transmitted in straight lines through the water sample. Turbidity in water is caused by suspended matter, such as clay, silt, finely divided organic and inorganic matter, soluble coloured organic compounds, plankton and other microscopic organisms. The clarity of a natural body of water is a major determinant of the condition and productivity of that system.

Van Dorn sampler - a messenger-operated water-suspended sediment point sampler used to collect samples at a specified depth. The long axis of the cylinder can be lowered either horizontally or vertically.

Volatile constituents - components of a sample which are readily lost by evaporation. They include dissolved gases as well as substances with low boiling points.

Watershed - all lands enclosed by a continuous hydrologic-surface drainage divide and lying upslope from a specified point on a stream (see also drainage basin).

Water pollution - the addition of harmful or objectionable materials to water in sufficient quantities to affect its usefulness adversely.

Water quality criteria - scientific information, e.g. concentration-effect data, used to recommend water quality objectives.

Water quality objective - a concentration or a narrative statement describing the water body, which, when met, will protect the uses of the water.

Water quality standard - the concentration of a constituent or a narrative statement describing the water body established under statutory authority, e.g. legally enforceable.

Zooplankton - microscopic animals living unattached in aquatic ecosystems. They include small crustacea, such as Daphnia and Cyclops, and single-celled animals, such as protozoa.

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